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Number 370

March | April 2021

nitrogen+syngas

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Turbulence in ammonia markets

Sustainable nitrogen production

Urea plant reliability

Safe handling of ammonia catalysts

NITROGEN+SYNGAS
ISSUE 370
MARCH-APRIL 2021

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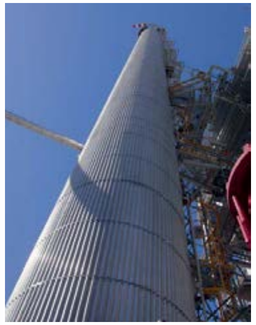

Cover: Urea reactor. Source: Saipem.

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Published by: BCInsight

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What about methanol?

The ammonia industry seems to have quite a buzz about it at the moment. As can be glimpsed in our Nitrogen Industry News section this issue, the number of proposed green ammonia production sites continues to grow, as does interest in ammonia as a hydrogen or energy carrier, while the shipping sector continues to seriously consider ammonia as a green fuel candidate for the longer term. The latter prospect could see current ammonia demand double by 2050, although as our article on sustainable nitrogen production on page 22 notes, whether enough renewable power will exist by then to generate that must be seriously doubted.

But while ammonia has been grabbing headlines, what about the other major syngas derivative, methanol? Methanol has also been touted as a clean, or at least cleaner burning fuel, with an unsuccessful move into methanol fuelled vehicles in 1990s California, and a far more successful uptake as a gasoline blendstock in modern China. But production of methanol has the potential to be a use for sequestered carbon dioxide streams, and green methanol can be a bridge to production of a wide variety of downstream carbon-based chemicals, from gasoline to dimethyl ether and polyolefins, all using existing technology. One straw in the wind is that this March, Swedish power to fuel company Liquid Wind joined the Methanol Institute. The company says that it is looking at combining captured CO_2 with electrolysis-generated hydrogen to produce carbon neutral fuels.

> **"We may be beginning to see the outlines of a new fuel and chemical paradigm"**

Methanol also has considerable potential as a shipping fuel, and one that might be available sooner and more easily than hydrogen or ammonia. AP Møller-Maersk recently announced that it will build the first carbon neutral ship powered by biomass-based green methanol by 2023; a 2,000 TEU bulk container ship. Elsewhere, Sweden's Alfa Laval is currently beginning the third stage of a major programme testing methanol's use in ships in Denmark, in conjunction with engine manufacturer MAN Energy Solutions and the Danish Technological Institute (DTI), as well as biofuel producer Nordic Green. Green methanol, the consortium says, could be burned in unmodified diesel engines, though it would require new engine software, which it hopes to develop as part of the test programme. German energy company Uniper also recently announced that it is launching a program designed to scale up green methanol as a sustainable and carbon-neutral marine fuel.

This flurry of activity in the shipping industry, following on from the publication of IMO Maritime Safety Committee interim guidelines on using methanol as a marine fuel has led the Methanol Institute to suggest that demand for methanol could grow fivefold over the next 30 years, reaching 500 million t/a by 2050 from its 2019 total of 98 million t/a – itself double the 2010 figure. In January the Methanol Institute published a report in conjunction with the International Renewable Energy Agency (IRENA) called Innovation Outlook: Renewable Methanol which "describes an ambitious, yet realistic, energy transformation pathway based largely on renewable energy sources and steadily improved energy efficiency." The report concludes that by 2050 that 500 million t/a of methanol production could be comprised of up to 135 million t/a of methanol from biomass (biomethanol); a further 250 million t/a of methanol from green hydrogen and captured CO_2; and 115 million t/a of conventional/legacy methanol made from natural gas.

It is my frequent refrain when discussing such projections that this kind of widespread uptake can only happen if the production costs of hydrogen from electrolysis fall dramatically. However, even an old cynic like me cannot help but be impressed at how far such costs have already fallen in just a decade, and there are no doubt further technology improvements to come which will reduce costs at the same time that economies of scale unlock further per tonne improvements in production economics, just as they did for gas- and coal-based production. It is possible that we may be beginning to see the outlines of a new fuel and chemical paradigm that will transform the 21st century in the same way that oil and gas did the 20th, and the good news for ammonia and methanol producers is that those chemical building blocks may well be at the heart of it.

Richard Hands, Editor

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Price trends

Market Insight courtesy of Argus Media

NITROGEN

In the ammonia market, another spate of spot deals has emerged in recent weeks at steep premiums to any previously done business in the spot market, pushing f.o.b. prices in the Black Sea and c.fr prices in the US Gulf to highs not seen since late-2015.

Severe supply tightness continued to dictate sentiment, and producers were able to lift offers significantly as spot cargoes became available. The deficit appeared to be most concentrated around the US, where following winter storm Uri in February, four production plants remained offline in the US Gulf, and some plants also remained offline in other regions following weather-related gas shortages.

Outside the US, Trinidadian producers were ramping up production as much as possible, but Nutrien's outage on the island continued, and the producer was forced to step into the market for more Algerian tonnes towards the end of February at a price netting forward to $470/t c.fr US Gulf. The spot sales added upward pressure to the Tampa settlement for March, with a huge increase on the previous month's $330/t c.fr expected.

Some producers are already reporting to be sold out for April. Price increases into May are widely expected. Argus' trade balance shows a surplus in the second quarter, with prices falling gradually from April as production normalises. Other market drivers included concerns over the US application season, a Pivdenny price jump of over $40/t, and an outage in Saudi Arabia.

On the urea side, freight has been the dominant topic in the market recently. Rates have risen further after the jump in late February, with Middle East-Brazil freight rates now quoted at $38-40/t, double the rate at the start of the year. The volatility and uncertainty created impeded new sales of urea.

Prices have moved up in Egypt and the US but remained stable elsewhere. Egypt benefitted from strong demand from Turkey in particular, while the US needed to buy substantial quantities of granular urea to arrive by May. Plants in the US were restarting after gas-related closures in February, but additional imports will be needed to cover the production loss.

Elsewhere, the demand outlook is very favourable for 2021. Increased grain plantings are forecast across the globe due to attractive prices. These are always weather dependent, but for now optimism abounds. The market is showing stability in many areas but some marginal gains in f.o.b. levels are likely in March, possibly affecting North Africa and the US. The urea market is forecast to swing to a surplus in the second quarter as demand wanes in the west and new capacity comes on line. Recent market drivers included expectations of agricultural demand, the supply situation in the US and global freight rates. ■

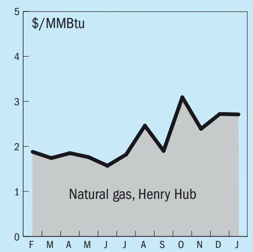
Table 1: Price indications

Cash equivalent	mid-Feb	mid-Dec	mid-Oct	mid-Aug
Ammonia ($/t)				
f.o.b. Black Sea	300-370	204-230	200-210	178-205
f.o.b. Caribbean	290-335	200-230	180-190	160-175
f.o.b. Arab Gulf	290-330	230-260	230-260	230-260
c.fr N.W. Europe	340-420	250-275	225-255	210-250
Urea ($/t)				
f.o.b. bulk Black Sea	300-380	230-250	230-245	230-253
f.o.b. bulk Arab Gulf*	350-380	260-290	249-270	264-285
f.o.b. NOLA barge (metric tonnes)	329-365	242-250	238	230-240
f.o.b. bagged China	320-365	270-300	265-290	275-295
DAP ($/t)				
f.o.b. bulk US Gulf	568-600	419-436	367-395	353-390
UAN (€/tonne)				
f.o.t. ex-tank Rouen, 30%N	157	157	157	157

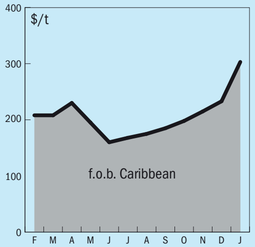
*Notes: n.a. price not available at time of going to press. n.m. no market. * high-end granular.*

END OF MONTH SPOT PRICES

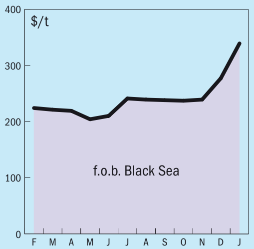
natural gas



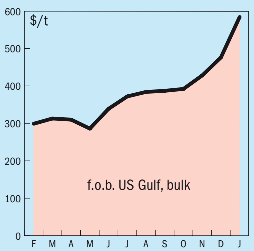
ammonia



urea

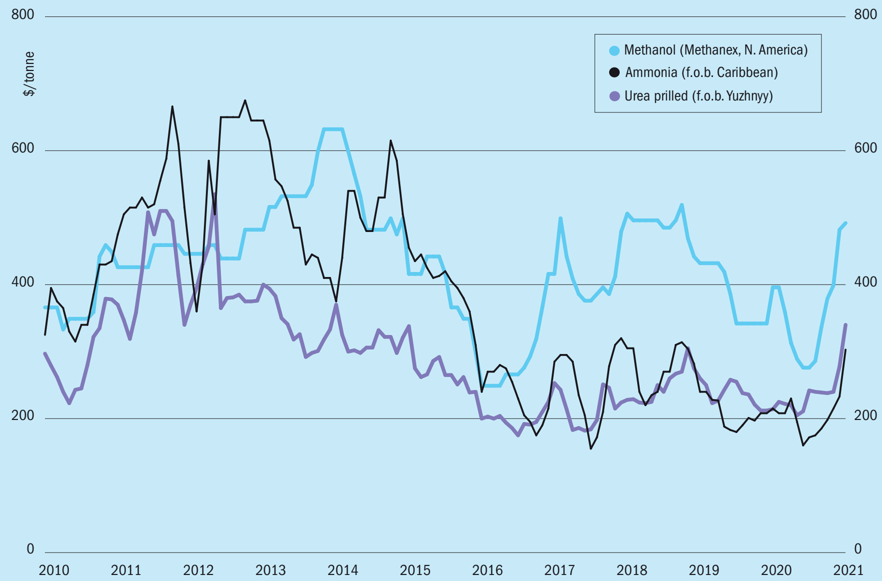


diammonium phosphate



Market Outlook

Historical price trends $/tonne



Source: BCInsight

AMMONIA

- The ammonia industry faced a difficult February, due to extremely cold weather conditions in the northern hemisphere. In the US, production outages resulting from winter storm Uri affected up to 7 million t/a of capacity.
- Supply tightness was exacerbated by ice in the Baltic Sea affecting deliveries from northern Russia, and gas curtailments in Trinidad and China. There were also unexpected production outages at EBIC in Egypt, and at the Burrup ammonia plant in Western Australia. As a consequence, ammonia prices rose rapidly to levels not seen for a couple of years.
- As the weather warms and plants restart, most of these restrictions will ease, but with many players in the market seeking emergency tonnages, some producers are already reporting to be sold out for April and price increases are expected at least until May, when returning supply is likely to bring prices back under control again.

UREA

- Freezing temperatures in the US also impacted upon urea production, with a number of producers forced to enter the market to secure supplies for customer contracts, driving up US prices dramatically. Spiking gas prices also encouraged producers to sell gas rather than make fertilizer.
- Urea demand continued in Turkey, helping to raise Egyptian prices, while increased freight rates have raised urea prices in many key markets, including Brazil.
- Expectations of an Indian tender in January-February did not materialise, but could still drive prices higher when it occurs, especially as supply from Middle Eastern suppliers remains tight. Buying from China and Brazil has also been subdued.
- However, expectations for 2Q 2021 are for a surplus in the market as demand wanes in the west and new capacity comes on line.

METHANOL

- Oil prices have been on an upward trend, beginning 2021 at around $50/bbl for Brent crude, and rising to $70/bbl by the start of March.
- Methanol prices have risen accordingly, assisted by news that gasoline and diesel consumption in China has continued to run at higher than pre-virus levels this year following the faster-than-expected return of factory activity and infrastructure building following the Lunar New Year holiday.
- However, there is also the possibility of increased methanol supply in China as the country emerges from winter and natural gas supply curtailments are eased for gas-based methanol producers. There are also expectations of increased supply from Iran.
- US supply was disrupted by the cold winter weather in the southern states and US Gulf, and Trinidad remains impacted by gas supply issues. The fire at Tjeldbergodden also removed supply from Europe during January. ■

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Nitrogen Industry News

DENMARK

MoU for power to ammonia facility

Copenhagen Infrastructure Partners (CIP) has unveiled plans to build Europe's largest power-to-ammonia facility at the Danish port of Esbjerg, based on electricity from offshore wind turbines. The company said the plant will consist of 1GW of electrolysis capacity, capable of supplying sufficient hydrogen to produce 300,000 t/a of ammonia, and that the ammonia will be used as both as agricultural fertiliser and as fuel for the shipping industry. Excess heat generated in the process would be used to provide heating for around one third of local households in communities around the plant, to be sited on the west coast of Denmark. The company has signed a memorandum of understanding for the project with companies from both the agriculture and shipping sectors, including Danish Crown, Arla, DLG, Maersk and DFDS Seaways. CIP anticipates that it would cost approximately $1.2 billion to build the facility. They are currently seeking investors for the project and expect that the investment decision would be reached by 2023. The plant could enter commercial operations in 2026.

Development partner Danish Crown, Europe's largest pork producer, says that by replacing fossil fuel derived ammonia with 'green' fertilizer, up to 1.5 million t/a of CO_2 emissions could be removed. Meanwhile shipping partners Maersk and DFDS are looking to decarbonise their fleets. Henriette Hallberg Thygesen, CEO of Fleet & Strategic Brands, A.P. Moller-Maersk said: "we consider green ammonia as a promising option for marine fuel and a dual-fuel engine for ammonia is under development. We are optimistic that ammonia, along with methanol and alcohol-lignin blends will be powering Maersk-vessels in the future." The company is targeting 2023 for its first ammonia powered vessel. ■

Topsoe reports strong growth

Haldor Topsoe's 2020 annual report has shown a 5% increase in revenue to $1.0 billion for 2020 in spite of the covid pandemic. Earnings before interest and taxes were up 6% to $123 million. Last year also saw the launch of a strategic transformation of the company, with the aim: "to be recognized as the global leader in carbon emission reduction technologies by 2024." This has led to a restructuring of the company focusing on three pillars. The first, Global Strategy & Innovation, combines the alignment of company strategy with research and development activities. The second, Global Commercial, combines all commercial activities in one global organisation to develop a strong commercial mindset, and enhance customer focus and service, with fast commercialisation of new product offerings. The third pillar, Global Supply, brings together the full supply chain activities to produce high-quality products more efficiently.

The company has also recently committed to the 'Science Based Targets initiative', led by the UN Global Compact, World Resources Institute, and World Wildlife Fund, to set short-term targets to reduce carbon emissions. Topsoe's short-term target is to reduce greenhouse gas emissions from its own production by 15% in 2021 compared to 2019.

SINGAPORE

Yara joins ammonia tanker project

The Maritime and Port Authority of Singapore (MPA) and fertilizer giant Yara International have joined previously existing partners Malaysian shipping line MISC, Lloyd's Register, Samsung Heavy Industries and MAN Energy Solutions in the ammonia-fuelled tanker joint development project (JDP). Yara will work alongside MISC, LR, SHI and MAN to develop ammonia powered ships while the consortium will use MPA's experience as a bunkering hub and flag state to gather insights on safety issues and ammonia bunkering procedures, as well as gaining access to research capabilities in Singapore. MISC says that it is aiming to be operating an ammonia-fuelled tanker by 2025.

"The addition of MPA and Yara means that the alliance which was first unveiled in January 2020, now has a complete representation from all areas of the maritime ecosystem. The experience and expertise of each partner will be central to the success of the initiative, from conception to project realisation,"

"Supporting the enabling role of ammonia in the energy transition, we recognize the need for value chain collaboration to make zero emission shipping by using ammonia as a fuel a reality," said Magnus Ankarstrand, EVP clean ammonia, Yara.

Last September, Lloyd's Register granted approval in principle to Samsung Heavy Industries for its ammonia-fuelled tanker design, a landmark project being carried out by MISC and engine manufacturer MAN Energy Solutions.

CHINA

Stamicarbon to provide urea plant license

Stamicarbon has signed a licensing and equipment supply agreement with Henan Xinlianxin Chemicals Group Co. Ltd for a second ultra-low energy grassroots urea plant in Jiangxi province. Henan Xinlianxin is currently commissioning the first plant, designed with Stamicarbon's *Launch Melt*™ low energy design. It represents the third licensing project in five years between the two companies, following a revamping project signed in 2016 and the award of the license for the first low energy urea plant design in 2017. Stamicarbon will deliver the process design package and proprietary high pressure equipment in *Safurex*®, together with associated services for both the urea melt plant and prilling plant. The urea plant, using a pool reactor, will have a capacity of 2,330 t/d, and is expected to start up in 2023.

New melamine plant licensed

Eurotecnica, the technology arm of Proman Group, has been awarded a contract by the Yankuang Group, one of the largest coal and chemical companies in China, for the licensing, engineering and procurement of Eurotecnica's 24th melamine plant based on its proprietary Euromel® technology. This project will be the fifth large scale single-train 60,000 t/a capacity plant award in a row, and will bring total Euromel nameplate capacity worldwide to 860,000 t/a, according to Eurotecnica. The new project will feature a single reactor arrangement and Eutotecnica's, zero pollution concept, as well as incorporating technological improvements for reduction of energy consumption.

SWITZERLAND

Clariant sets science-based climate targets

Clariant has committed to new 2030 targets to reduce CO_2 emissions in line with the Science Based Targets initiative (SBTi). The new targets, which have been officially validated by the SBTi, set out ambitious absolute emission reductions in the company's operations and supply chain. Between 2019 and 2030, Clariant is aiming for a 40% absolute reduction in scope 1 and 2 greenhouse gas emissions and a 14% absolute reduction in scope 3 greenhouse gas emissions, equivalent to the level of decarbonisation required to keep global temperature increase below 2°C.

Alongside the CO_2 emissions targets will be other targets covering of water intake, waste water, hazardous waste, landfilled non-hazardous waste, and nitrogen oxide emissions (NOx). Clariant has also committed to the implementation of sustainable water management systems at all sites in areas of high water stress by 2030. The company has also adapted important governance aspects to steer its transformation, such as linking greenhouse gas emissions reduction to remuneration and implementing internal carbon pricing. The company says it is aiming to reduce emissions by focusing on increasing energy efficiency through digitalisation of its operations, increasing use of green electricity and low carbon fuels, and accelerating innovation in value chains with increased use of low carbon raw materials such as waste streams and sustainable bio-based materials.

"Sustainability is one of Clariant's five strategic pillars and guides the path of our transformation into a leading specialty chemicals company. Our new targets reflect our commitment and responsibility toward enabling a more sustainable future. Innovation and transformation are the two vital elements that will lead us to success," said Bernd Hoegemann, Chief Transformation Officer at Clariant.

GERMANY

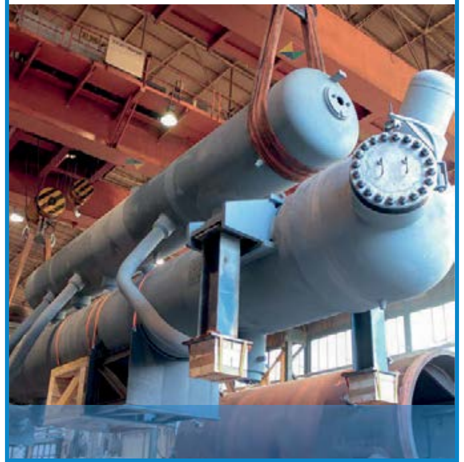
BASF and Siemens Energy to cooperate on carbon management

As part of a strategic partnership, BASF and Siemens Energy plan to accelerate commercial implementation of new technologies designed to lower greenhouse gas emissions. By combining BASF's technological expertise with Siemens Energy's innovative product and services portfolio, BASF aims to extend its leading role in lowering CO_2 emissions in chemical production. Several pilot projects at its Ludwigshafen site are under discussion. BASF's headquarters is one of the largest chemical production sites in the world.

Possible pilot projects include the construction of a PEM (proton exchange membrane) electrolyser for hydrogen production with an output of 50 MW with the possibility of modular capacity expansions and the installation of a high-temperature 50 MW thermal heat pump for generating process steam from waste heat in a production plant. In addition, modernisation of the power grid at the Ludwigshafen site using digital and CO_2-optimised products from Siemens Energy is being evaluated. A study is also underway to assess the potential for common system and catalytic converter development in an effort to boost the efficiency of electrolysis plants and for collaboration in generating electricity from wind energy.

Dr. Martin Brudermüller, Chairman of the Board of Executive Directors of BASF SE said: "by cooperating with Siemens Energy, we stand to benefit from the expertise of a first-class partner for implementing our carbon management, a partner with whom we can accomplish projects of a commercial scale. At BASF, we want to develop and implement new low-CO2 technologies as quickly as possible. If we want to use such technologies on a large scale, we will need appropriate regulatory framework conditions and tar-

BORSIG



↘ WASTE HEAT RECOVERY SYSTEMS

↘ WASTE HEAT BOILERS FOR AMMONIA PLANTS

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geted support. We need renewable electricity in large quantities for this, and we need it at competitive prices."

Topsoe to collaborate on green ammonia facility

Haldor Topsoe has signed a memorandum of understanding with AQM Capital LLC ('Aquamarine') to build a green ammonia facility in Germany. The first planned stage will use Topsoe's proprietary solid oxide electrolyser cells (SOEC) to produce green hydrogen from 100 MW of renewable electricity. Topsoe says that SOEC offers up to 30% more hydrogen output compared to standard technology such as PEM and alkaline electrolysis. The hydrogen will then be processed into 300 t/d of green ammonia, also using Topsoe technology. Aquamarine will develop the project and seek relevant permits, which will be located in northern Germany close to existing offshore wind farms, where the product can be sold to the marine shipping industry. Subject to a final investment decision, the facility is expected to be commissioned in 2024.

"We look forward to our partnership with Aquamarine on our SOEC and ammonia technology. Driven by our vision to be recognised as the global leader in carbon reduction emission technologies by 2024, we are excited by our low-carbon solutions and attractiveness for our customers. This project is innovative in both its use of cutting edge technology and its scale and will lead the way," said Amy Hebert, Chief Commercial Officer, Topsoe.

"We are delighted to be working with Haldor Topsoe toward the commercial application on its cutting edge SOEC technology to bring green products to market as part of the global move toward decarbonization. We expect to be soon announcing other partners who will be joining the effort, as we move forward to build HydrGEN into a global green hydrogen products leader," said Joel H. Moser, founder and CEO at Aquamarine.

NORWAY

Yara secures partners for Porsgrunn green ammonia project

Yara International has brought in two partners to help deliver the switchover to green ammonia production at its Porsgrunn production plant in Norway. The company signed a letter of intent on 18th February with Norwegian state-owned Statkraft, Europe's largest renewable energy producer, and investment firm Aker Horizons.

Yara first unveiled its ambitious project to fully electrify the Porsgrunn plant and produce 500,000 t/a of green ammonia at the end of last year (*Fertilizer International* 500, p10). This could be delivered within 5-7 years, according to Yara, provided enough renewable power was available and public co-funding was secured.

"With Statkraft and Aker Horizons onboard we gain key expertise within renewable electricity, power markets, industrial development and project execution, giving us a unique opportunity to realize the project," said Svein Tore Holsether, President and CEO of Yara.

Yara and its new partners will be targeting growing market opportunities for both green ammonia and green hydrogen in areas such as agriculture, shipping and industry. These will be pursued through the recently created Yara Clean Ammonia business unit.

"A partnership with Yara and Statkraft, two fellow Norwegian industrial pioneers, marks the beginning of a new industrial adventure in Norway," said Oyvind Eriksen, chairman of Aker Horizons. "The first project in Porsgrunn can be a lighthouse project – providing competitive advantage in a growing global hydrogen economy and building on existing capabilities in the Norwegian supplier industry to create new jobs for the future."

UNITED KINGDOM

AIC responds to DEFRA urea consultation

The UK Agricultural Industries Confederation (AIC) has responded to proposals by the UK government's Department of the Environment, Food and Rural Affairs (DEFRA) to reduce ammonia emissions from farming by restrictions on the use of urea fertilizer, one option of which would be a ban on the use of urea in the UK. The AIC said in its response that a ban on the sale and use of solid urea fertilizer is unwarranted. Instead, the organisation has urged the government to give the fertilizer sector the opportunity to reduce ammonia emissions through an industry-led commitment, based around the principles of nitrogen use efficiency (NUE), which will involve demonstrating delivery through a combination of FACTS qualified advice and farm assurance auditing.

"This industry-led approach would have many advantages over banning a nitrogen source from use," said Robert Sheasby, AIC's Chief Executive. "With an end goal to achieve UK 2030 emissions targets, and mindful of all that livestock farmers will need to do on the manure management front, the fertilizer sector is stepping up to deliver its part in curbing ammonia emissions from urea-based fertilizers."

The AIC and partners across the industry and food chain have committed to improving farm nutrient balance (notably for nitrogen and phosphorus) and NUE by 2030.

"We recognise that all fertilizer sources have their pros and cons, and that farmers and growers need the right products, for the right weather conditions, at the right time. This is where the fertilizer sector and all professional advisers are best placed to help. By supporting farmers to improve their overall crop NUE it's a win-win situation," added Sheasby.

IRAN

Lordegan urea plant begins exports

Iran's Lordegan Petrochemical Company, based at Chaharmahal in Bakhtiari province, says that it has exported its first ammonia shipment to Turkey. The 2,500 tonne cargo has been despatched to the neighbouring country, and Lordegan says that its first urea cargo is expected to be exported soon. The recently completed Lordegan plant has a capacity of 670,000 t/a of ammonia and over 1.0 million t/a of urea.

NETHERLANDS

Sustainable urea production

A consortium led by the Netherlands Organisation for Applied Scientific Research (TNO) has received a grant of €21 million from the European Commission under the Horizon 2020 Framework Program to investigate and develop the potential of industrial symbiosis to convert residual gas emissions from steel production into resources for urea production. TNO will be partnered in the project by Maire Tecnimont subsidiaries Stamicarbon, MET Development SpA and NextChem SpA.

The "Initiate" (Innovative Industrial Transformation of the steel and chemical industries of Europe) project will demonstrate a novel, symbiotic and circular process that transforms residual steel gases into resources for urea production. The core of this process is a modular carbon-capture utilisation-and-storage (CCUS) technology, integrating the flexible conditioning of time-dependent and carbon-rich steel gases with the synthesis of ammonia. Once a successful technology pilot unit is completed, the project partners aim to scale up to a 150 t/d urea demonstrator plant by 2026.

Stamicarbon will be responsible for the commercial implementation plan. The main objective of the demonstration plant is to justify the viability and prove the capability to produce ammonia, while in the next phase the commercial implementation plan is focused to establish an industrial-scale Initiate plant reference for the production of urea.

CROATIA

Petrokemija completes repairs and revamp

Croatian fertilizer producer Petrokemija has completed a $14 million programme of repairs and revamps at its Kutina chemical site. The revamp included $3.5 million of investment at the company's ammonia plant to install a new air preheater for the primary reformer. Other works were conducted on the urea plant, water processing and power facilities, sulphuric acid, ammonium nitrate, CAN and NPK plants. Production at the site resumed on February 8th, after damage caused by a 6.4 magnitude earthquake in Petrinja in central Croatia on December 29th shut the site down over safety concerns. Repairs to the AN/CAN plants continue and the plants will re-start soon, according to the company.

JAPAN

Jera to produce ammonia for power

Jera, a joint fuel-procurement venture between Japan's Tokyo Electric Power and Chubu Electric Power, has signed a memorandum of understanding with and Malaysian oil, gas and fertilizer giant Petronas to develop CO_2-free ammonia production using renewable energy, including hydro-electric power. Jera aims to use the ammonia as part of its plan to decarbonise electrical power generation. The consortium accounts for about 10% of Japan 's total emissions of carbon dioxide, via its gas- and coal-fired power plants. It aims to reduce this to near-zero by 2050 via use of renewable hydrogen and ammonia.

The companies plan to begin a demonstration project within the next year in which coal and ammonia will be mixed and used as fuel at a thermal power plant in Aichi prefecture in central Japan. This will provide the companies with more knowledge and will support their move towards creating a power generation facility that will burn only ammonia. While renewable ammonia will be more costly as a fuel than coal, Jera says that it aims to reduce costs by taking charge of every process, from development to procurement to power generation. Japan is pushing hard with ammonia fuel development. The Ministry of Economy, Trade and Industry has set a target of introducing 3 million t/a of ammonia fuel by 2030.

INDIA

Government to sell 20% stake in NFL

The Indian government has decided to sell 20% of its existing 74.7% stake in state-owned National Fertilizers Ltd (NFL) via an offer for sale. The Department of Investment and Public Asset Management has invited tenders from merchant banks for the sale process. NFL is the second largest producer of urea in India, with a share of about 15% of total domestic production. The sale of the 20% stake in National Fertilizers Ltd is expected to bring in around $54 million for the government. ■

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Syngas News

NIGERIA

Plan for large scale methanol production

The Nigerian National Petroleum Corporation (NNPC) says that it plans to build a $3 billion methanol plant on Brass Island in the Niger Delta to produce up to 10,000 t/d of methanol using from gas supplied by Shell. A final investment decision was made by NNPC, DSV Engineering and the Nigerian Content Development and Monitoring Board, a state agency set up to ensure Nigerian involvement in oil and gas projects. Around 70% of funding for the project will come from international lenders, including the China Export-Import Bank, the African Development Bank and international commercial banks, with the rest funded from an equity issue. BP has signed a 10 year offtake deal for the plant's output with the Brass Fertilizer & Petrochemical Company, the entity set up to operate the plant. Construction of the plant is expected to be completed by 2025.

The Nigerian government is hoping to aggregate and monetise 'stranded' gas in the Brass Island area, where reserves amount to 10 tcf of gas. The methanol will mainly be for export, though it will also replace all of Nigeria's 50,000 t/a or so of methanol imports.

NORWAY

Tjeldbergodden methanol plant back onstream

Equinor's methanol plant at Tjeldbergodden has returned to production following a fire at the plant on December 2nd last year. Equinor says that the fire broke out in the compressor building of the methanol plant, causing the plant and associated air separation unit to be shut down. No-one was injured in the incident. An investigation into the causes of the fire is still under way.

"Our number one priority has been safe and secure start-up of the facility," said Lena Skogly, plant manager at Tjeldbergodden. "I'd like to thank everyone who contributed to get the facility back on line, both our own employees and external expertise. It takes time to check all technical matters and damage due to the fire. Thorough inspections in the facility, as well as testing and validation of equipment that may have been affected by the fire have been carried out. During these efforts, it's been important to extract learning from the incident and ensure that we have the technical and operative barriers in place before start-up. This painstaking work makes us confident that the facility will produce in a safe and stable manner."

Clean hydrogen project

INOVYN, a subsidiary of INEOS, plans to build a new clean hydrogen supply hub at its manufacturing site in Rafnes. The company will build a 20 MW electrolyser to generate clean hydrogen powered by zero-carbon electricity for the Norwegian transport sector. INOVYN is aiming to eventually establish a network of refuelling stations in Norway to provide buses, trucks and taxis with clean hydrogen, and is targeting producing enough hydrogen to fuel up to 400 buses or 1,600 taxis.

NEW ZEALAND

Methanex to close Waitara Valley

Methanex has announced that it plans to close one of its methanol plants in New Zealand, at Waitara Valley, Taranaki, New Zealand, because it has not been able to secure sufficient gas feedstock to keep the facility operating. Gas production from the Pohokura field which feeds the plant has been declining, and the covid pandemic has delayed new drilling and exploration by gas producers in the region. The company said that it would lay off staff and keep the facility on a care and maintenance basis in a safe condition so that it could be restarted should gas become available. Methanex will continue to operate its two methanol plants at Motunui. Waitara Valley has a capacity of 500,000 t/a of methanol, and the two Motonui plants a further 1.7 million t/a of production capacity.

UZBEKISTAN

Technology licensed for MTO project

Versalis, the chemical arm of Italian energy major Eni, has licensed a low density polyethylene/ethyl vinyl acetate unit to be built as part of a new gas to chemical complex based on methanol to olefins technology in the Karakul area of Uzbekistan. Enter Engineering Pte. Ltd. will act as licensee on behalf of Jizzakh Petroleum JV LLC, who will own and operate the LDPE/EVA unit and the entire gas to chemical complex once built.

The contract award follows the September 2020 confirmation of Jizzakh Petroleum as a partner in the MTO gas-to-chemicals project, formed in May 2019 via an agreement between Uzbekneftegaz, JSC Uzkimyosanoat, Enter Engineering, and Air Products and Chemicals Inc. Polyolefin capacity will be 500,000 t/a.

GERMANY

Porsche looking to low carbon MTG as a vehicle fuel

Dr. Frank Walliser, Porsche vice president of GT cars and motorsport, has said in a recent interview that cars running on synthetic fuel could deliver an 85% reduction in carbon emissions compared to fossil fuel equivalents, representing lower "well to wheels" emissions than an electric car, once emissions related to manufacturing are factored in. A synthetic fuel should be ready for testing in 2022, Walliser said, adding that this fuel could be used in all of Porsche's current internal-combustion engines without modifications. The fuel will come from a pilot plant in Chile named Haru Oni, being developed by Porsche and Siemens, which will use wind-generated electricity to electrolyse water into hydrogen and oxygen. The hydrogen will then be combined with carbon dioxide from the air to produce methanol, which in turn can be used to create synthetic gasoline, diesel, or kerosene aviation fuel. Porsche says the plant would produce 130,000 litres (96 tonnes) of fuel by 2022, ramping up to 55 million litres per year (40,000 t/a) by 2024, and 550 million litres per year (400,000 t/a) by 2026. Approximately 40% of the fuel produced will be gasoline, of which Porsche will be the primary consumer initially.

JAPAN

Cow manure to methanol project

A pilot plant is being developed in Sapporo, Hokkaido to convert urine and feces from thousands of dairy cows into methanol via a biogas process developed at Osaka University. Industrial gas supplier Air Water Hokkaido Inc. and contractor Iwata Chizaki Inc., both based in Sapporo, are partnering with the university to establish the plant. Methanol from the process will be used locally as fuel, replacing heavy fuel oil, and will also be used to power fuel cells.

The process dissolves methane from biogas in a transfer fluid and exposes it to ultraviolet light, converting it into methanol and formic acid at normal temperatures and pressures, without production of carbon dioxide. Single pass methanol conversion efficiency has been improved to 14%. The formic acid can be used as an additive for cattle feed, or a source for hydrogen production. The current trial facility is to be expanded into a pilot plant by 2024, and could be rolled out to other areas in and outside Hokkaido by 2030.

AUSTRALIA

Plans for large scale renewable hydrogen plant

Australian natural resources company Province Resources says that it is looking to produce renewable hydrogen from 1GW of wind and solar energy in the Gascoyne region of Western Australia. Primarily a mining company, Province recently acquired Ozexco, which holds exploration license applications for minerals such as salt and potash in the region. The proposed renewables development, HyEnergy, as well as a parallel mineral prospecting plan, would be located in a flat desert area near the town of Carnarvon, where Province Resources aims to create a hydrogen hub, eventually making up to 60,000 t/a of hydrogen and 300,000 t/a of ammonia, for sale to Japan and other Asian markets. In a first stage, a pilot hydrogen and ammonia plant is envisaged at Carnarvon. The company also says that it plans to investigate the potential for the blending hydrogen into the Dampier-Bunbury natural gas pipeline.

INDONESIA

Is coal gasification the way forward for Indonesia?

As the world increasingly turns away from burning coal for power, Indonesia, the world's largest exporter of thermal coal, is left considering what to do with its abundant coal reserves. The answer may be provided by a number of recent project announcements for coal gasification projects. In December, state-owned Tambang Batubara Bukit Asam, also known as PTBA, announced a $2.1 billion coal to methanol and downstream dimethyl ether (DME) project in partnership with state oil and gas major Pertamina and US-based Air Products and Chemicals. Construction is due to begin on the new plant in mid-2021 near PBTA's coal mine at Tanjung Enim on South Sumatra Province. Commercial operations are targeted to start in 2025, with a production capacity of 1.4 million t/a of DME, using 6 million t/a of coal as feedstock – around 20% of PTBA's annual output. The DME would be used as an LPG blendstock or substitute, although questions have been raised about its potential cost compared to imported LPG.

Now Bumi Resources, Indonesia's largest coal producer, has announced its own coal gasification project. Its subsidiary Kaltim Prima Coal is looking to partner with Ithaca Resources and – once again – gasification technology supplier Air Products to build a coal-to-methanol facility in East Kalimantan, consuming another 8.7 million t/a of coal. Air Products would invest about $2 billion "to build, own and operate" the facility, could be up and running as soon as 2024. They intend to supply the methanol to the government's expanding biodiesel program. Bumi is also conducting a pre-feasibility study for a second coal-to-methanol project with a potential on-stream date of 2025. Bumi is aiming to settle a $1.7 billion debt in 2022 and conclude a mandatory bonds-to-equity conversion by 2024 following its debt restructuring process in 2017.

Indonesia's other major coal company, Adaro Energy, is also looking at a coal gasification project, again producing methanol, or potentially methanol to olefins. There are also underground coal gasification (UCG) pilot projects which would generate syngas being developed by Kideco Jaya Agung, Indominco and Medco Energi Mining Internasional.

DENMARK

Topsoe building electrolyser manufacturing facility

Haldor Topsoe says it will invest in a manufacturing facility to build large-scale solid oxide electrolyser cell (SOEC) electrolysers to meet future customer needs for green hydrogen production. The facility will produce electrolysers with a total capacity of 500 MW per year, with the option to expand to 5 GW per year. Topsoe claims that its proprietary SOEC electrolysers offer superior performance in electrolysis of water into hydrogen, compared to standard alkaline or PEM electrolysers. The facility is expected to be operational by 2023.

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OBITUARY

Alan Heywood

Bodo Albrecht, BASF writes: Among the many sad news of 2020 is the passing of Alan Heywood, a leading authority in the advancement of nitric acid catalysts and recovery systems. At the time I first met Alan in 1997, he already had 6 patents to his name ranging from improved aircraft gas turbine igniters and "ZGS" grain stabilized precious metal composite materials to PGPs "cartridge" catalyst, arguably the first true catalyst system ever produced.

To label Alan as a creative metallurgist would, however, miss the mark. Like few others, Alan was relentless in constantly optimizing the products he was concerned with, thereby helping to make the companies he represented stand out from the crowd. With the cartridge catalyst, for example, came the "stretcher", a device making catalyst exchanges quick and easy. The product became a cornerstone to the success of what then was PGP, a company that launched an office in the UK and a precious metals plant in Shannon, Ireland, under his leadership. It also became famous for another one of his creations, a highly customized chemical catalyst branded as "Silocat".

At the time, back in the '90s, nitric acid catalysts were sold by the troy ounce, discouraging innovation since any kind of cost savings to the customer also reduced revenue. The catalyst system, often integrated with platinum recovery features and even the refining built in the price, changed all that allowing for continuous product improvement and convenience to customers while incentivizing the innovators.

Joining me at what was Degussa back in 1997 meant gaining access to the company's latest three-dimensional knitting technology. It was like a dream come true for him: the technology allowed for nearly limitless opportunities to evolve his "cartridge" concept using different knitting patterns, metal alloys and even wire diameters for each layer of the product. Alan's forensic intellect, sharpened by having worked with the aircraft industry prior to nitric acid, guided the team around him to look at these new designs from every conceivable angle to ensure safe operation, and high efficiency. "If you allow it to go wrong, it will" was his maxim, and it explains Alan's pedantic approach to quite literally putting every part of a new catalyst system under a microscope. I remember a time back in the 90s when he and I travelled with a metal guillotine to cut out precise samples from spent catalysts at customer sites, directly upon removal from the reactor. Sadly, the TSA eventually put an end to this.

Having grown up in the UK under the inspiration of a father who excelled at fixing mechanical devices of all kinds, Alan developed a lifelong passion for cars (the 'imperfect' kind from Italy, preferably – I never managed to convince him of the 'boring' perfection of Made in Germany), aircrafts, clocks and guns, and the beauty of the components they were made of. Some of you may also recall his genuine interest in the people he met, inquiring about the places people were from, and details of their lives.

Alan passed away in December of last year after having battled cancer for several years, growing weaker and weaker over time but refusing to give up. He is leaving a void to his family, his friends, and to the nitric acid industry which he impacted like few other people have. ■

Calendar 2021

! The following events may be subject to postponement or cancellation due to the global coronavirus pandemic. Please check the status of individual events with organisers.

APRIL

26-28 POSTPONED TO AUTUMN 2021
Syngas 2021,
BATON ROUGE, Louisiana, USA
Contact: Betty Helm, Syngas Association, Baton Rouge, Louisiana
Tel: +1 225 706 8403
Web: www.syngasassociation.com

MAY

26-27
Syngas Nitrogen Russia and CIS,
MOSCOW, Russia
Contact: Milana Stavnaya, Programme producer, Vostock Capital
Tel: +7 499 505 1 505
Email: MStavnaya@vostockcapital.com

JUNE

DATE T.B.C.
Argus East Europe Fertilizer,
Location T.B.C.
Contact: Argus Media
Tel: +44 (0) 20 7780 4340
Email: conferences@argusmedia.com

3-4
NH3 Event, ROTTERDAM, Netherlands
Contact: NH3 Event Europe
Tel: +31 10 4267275
Email: info@nh3event.com

22-24
China International Fertilizer Show,
SHANGHAI, China
Contact: CCPIT Sub-Council of Chemical Industry
Tel: +86 10 8425 5960
Email: zhengyingying@ccpitchem.org.cn
Web: en.fshow.org

28-30
IFA Annual Conference, LISBON, Portugal
Contact: IFA Conference Service,
49 Avenue d'Iena, Paris, F75116, France.
Tel: +33 1 53 93 05 00
Email: ifa@fertilizer.org

AUGUST

29-SEPTEMBER 2
65th AIChE Annual Safety in Ammonia Plants and Related Facilities Symposium,
SAN DIEGO, California, USA
Contact: Ilia Kileen, AIChE
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Plant Manager+

Preventing safety risks with a proper leak detection system

Several safety risks threaten urea high pressure equipment such as high pressures, high temperatures, various kind of corrosion phenomena, crystallisation risks, and the release of large volumes of toxic ammonia in case of a leak. A significant number of serious incidents with high pressure urea equipment still occur in the industry and, in 50% of cases, a failing leak detection system was one of the main causes. UreaKnowHow's Risk Register for a 316L urea grade reactor identifies 50+ safety risks of which 75% can be prevented by operating a proper leak detection system. In this article, UreaKnowHow answers some key questions about the importance of an effective active leak detection system.

Why is a proper leak detection system needed?

High pressure urea equipment has a pressure-bearing, carbon steel wall, which is protected against corrosion by a thin corrosion-resistant layer of stainless steel or duplex steel. This protective layer can be an overlay welding or a loose liner. Fig. 1 shows the installation of a stainless steel loose liner in a urea reactor.

A proper leak detection system is needed at all locations where a gap exists between the stainless steel or duplex alloy protection and the carbon steel, pressure-bearing parts. Any leak in the loose liner will lead to a dangerous situation where a large surface of the carbon steel pressure-bearing wall underneath the loose liner will be exposed to extremely corrosive ammonium carbamate. Real life experiences have proven that ammonium carbamate can corrode carbon steels with very high corrosion rates up to 1,000 mm (40 inches) per year. It is therefore imperative to have a detection system present that provides warnings of leaks at an early stage, i.e. active leak detection is required.

When there is a leak, crystallisation of the leaking medium is highly likely and will lead to clogging of the leak path. Once clogging occurs, there may no longer be any warning signs that there is a leak. This is an extremely dangerous situation as the corrosion of the carbon steel pressure-bearing wall will continue and can result in a sudden rupture of the vessel.

UreaKnowHow.com has collected and analysed more than 130 serious urea incidents. Based on this analysis, one can easily conclude that the number one safeguard for any urea plant is a proper leak detection system for loose liners of high pressure equipment.

Why is a passive leak detection system no longer acceptable?

In a passive leak detection system an indication of a leak in the liner only shows up when the leak emerges from the leak detection hole, or in case the leak detection hole is connected via tubing to, for example, a bottle with a phenolphthalein solution, when the colour of the liquid in the bottle changes.

There is a time delay before the leak is detected, i.e. when the leak emerges at the leak detector or when a field operator reports a leak. The latter case is even worse as the time delay can be much longer. There is a real risk that the leak detection path will get clogged, posing a serious threat to the integrity of the carbon steel, pressure-bearing wall.

A much more critical situation arises when clogging occurs in the tubing or underneath the liner and no solids are visible. Nobody will be aware of an extremely dangerous situation.

A leak contains ammonium carbamate, water and in some parts of the equipment also urea. At the lower pressure present

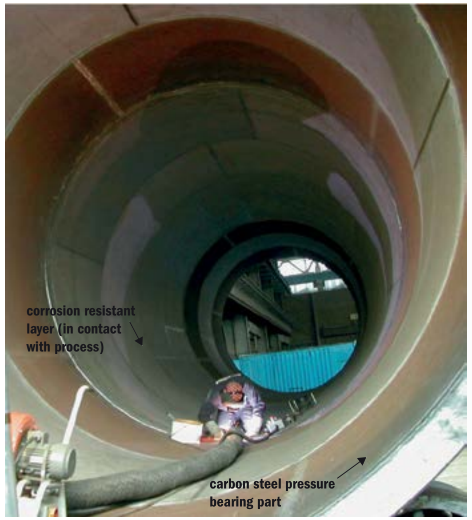


Fig. 1: Installation of a stainless steel loose liner in a urea reactor.

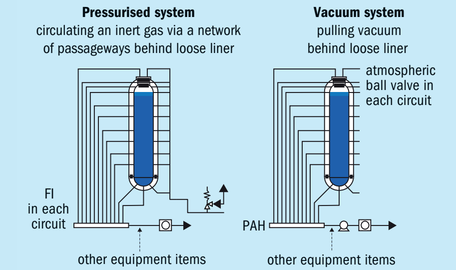
in the leak detection system, ammonium carbamate will dissociate into ammonia and carbon dioxide, but only if the temperature is higher than 60°C (140°F). Urea, however, can easily crystallise or polymerise and clog the leak detection path.

Avoiding the risk of clogging is the major challenge for any leak detection system. In case of clogging, the pressure and temperature increase prevents the dissociation of ammonium carbamate into the less corrosive components ammonia and carbon dioxide and the trapped ammonium carbamate causes very high corrosion rates for carbon steel. Under these conditions, ammonium carbamate can also actively corrode stainless steel after a period of time due to lack of oxygen for passivation.

Why is flushing with steam or condensate NOT a good practice?

To check that a leak detection system is not experiencing any clogging, or to unclog a leak detection system, some plants practice a flushing procedure with steam or water. This is not recommended and even dangerous! Introducing water can lead to corrosion. Corrosion is an electrochemical reaction so corrosion can only

Fig. 2: **Active leak detection systems**



Source: UreaKnowHow.com

take place when water is present. Without water there is no corrosion. The corrosion product will block the passageways and can also block the leak detection holes. A more threatening form of corrosion that can occur is stress corrosion cracking of the carbon steel pressure vessel wall, like hydrogen induced stress corrosion cracking or (bi-)carbonate stress corrosion cracking that can occur when solids, consisting of a mixture of ammonium-carbamate, urea and biuret, are present behind the liner in combination with water. Other contaminants like nitrates can also cause stress corrosion cracks in the carbon steel wall. Be aware that these cracks are not visible, they are behind the liner; nor can they be easily detected by the usual inspection techniques.

What types of active leak detection systems exist?

There are two types of active leak detection systems: a pressurised system, in which an inert carrier gas flows through the gaps underneath the liner and a vacuum-based system, where one pulls vacuum pressure behind the liner (see Fig. 2).

In a pressurised system an inert carrier gas circulates via machined grooves through the various leak detection circuits (compartments). Typically, each circuit includes a flow indicator with a low flow alarm. All circuits of one equipment item are combined on an equipment header. Sometimes the grooves are machined in a stainless steel buffer layer. In UreaKnowHow's opinion this does not provide a solution as stainless steels can show accelerated corrosion rates as described above, plus one cannot be assured that corrosive ammonium carbamate fluid is not present outside the grooves which will still corrode the carbon steel, pressure-bearing wall.

A vacuum system pulls vacuum behind the liner by means of a vacuum pump. That means that the whole area underneath the liner is under vacuum pressure. In the suction side of the vacuum pump a pressure transmitter with a high pressure DCS alarm is present. In this type of system all circuits of one equipment item are also combined on an equipment header.

Why choose a vacuum system?

UreaKnowHow recommends a vacuum-based leak detection system for several reasons. One reason is that when using a vacuum system there will be no risk of liner bulging and damage due to too high pressure in the leak detection system behind the liner. Some vendors try to avoid this risk of bulging by switching off the leak detection system when the pressure in the synthesis section is below a certain value. However, the drawback of this is that during these situations (heating up and cooling down of the synthesis section) one puts the highest (tensile) stresses on the liner welds and it is therefore important to have a properly working leak detection system during this phase.

A second important reason for recommending a vacuum-based leak detection system is because it provides direct detection of the entire carbon steel surface. A liner compartment is defined as being the part of the liner between four welds connecting the lining material to the carbon steel of the pressure vessel wall. In an active vacuum leak detection system, one is assured that the entire carbon steel surface underneath the loose liner of a liner compartment will be reached because the total area is under vacuum pressure and ammonia from any location will be directly pulled to the analyser.

How can a vacuum system distinguish a false indication?

Active leak detection systems consist of a network of gas-tight, high quality, instrumentation stainless steel tubing, but even gas-tight tubing may leak to a certain extent depending on the quality of the connections. In case of an active vacuum system, the actual vacuum pressure depends on the result of the sum of all leaking connections on one side and the capacity of the vacuum pump on the other side. Typically, the vacuum pressure will be in the range of 0.2 to 0.5 bara depending on the quality of the installation. In addition to continuously monitoring the vacuum pressure, an active vacuum leak detection system also has a continuous ammonia analyser. The AMMO LASER Leak Detection System, developed by UreaKnowHow.com and KeyTech Engineering Company, makes use of a very reliable, accurate, self-calibrating and multi-purpose ammonia analyser and provides an alarm at an ammonia level of 100 ppm.

An ammonia cloud in the atmosphere will most probably be sucked into the vacuum leak detection system, but will not penetrate all of the leak detection connections of the tubing. That will only be the case in the unlikely situation that the ammonia cloud covers the entire high pressure urea synthesis section. It is far more likely, therefore, that an ammonia cloud will penetrate only some of the connections while all other connections suck in air with hardly any ammonia. This situation will result in a small increase of the ammonia level from 2-3ppm to about 10-20ppm. The DCS operator will then be notified of an ammonia leak in the synthesis section, which can happen, for example, if there is a leaking flange connection or a local small ammonia cloud as a result of taking a sample. Subsequently one is able to take proper actions. After the leak has been eliminated, the ammonia level in the leak detection system will decrease and return to the normal background ammonia level in the atmosphere in the urea plant (e.g. 2-3 ppm). A liner leak will cause leaking fluid in the leak detection system to be sucked by the vacuum pump to the ammonia analyser. The leaking fluid contains minimum 30 wt-% ammonia (note that ammonium carbamate dissociates into ammonia and carbon dioxide). Even a small liner leak will quickly result in an ammonia level of 100 ppm and thus an alarm will sound. Furthermore, a liner leak will slowly increase in size due to corrosion and erosion and this will result in a gradual and continuous increase of the ammonia level. In this way, the AMMO LASER Leak Detection System can distinguish a false indication caused by an ammonia cloud in the atmosphere from a real liner leak.

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A turbulent year

Shutdowns in technical ammonia capacity due to the pandemic have contributed to make 2020 a turbulent year for ammonia markets, but plant shutdowns, strong fertilizer demand and a shortage of new merchant ammonia capacity are contributing to finally removing the burden of overcapacity.

PHOTO: NFC

Left: Ammonia storage at NFC, India.

Ammonia remains the second largest industrial chemical by tonnage, and global production and consumption continues to rise. As Table 1 shows, in 2019 this reached 183 million t/a, which was up 2% on the previous year, but CRU estimates that ammonia production rose by 4% to 190 million t/a in 2020 in spite of the impact of Covid. As a result, ammonia utilisation rates have been climbing from the low point of 79% that they reached in 2017-18 back towards 83%.

Since most ammonia is consumed at the point of production in the downstream manufacture of urea, nitric acid or various nitrates, the merchant market for ammonia remains only a small fraction of this total, however, representing around 11% of production, or 19.6 million t/a in 2019 (itself down 0.4 million t/a on 2018), and the full figure for 2020 is likely to be much the same as for 2019 or slightly lower (19.5 million t/a) in spite of the rise in overall ammonia production – most of the new ammonia production in 2020 was used in downstream urea plants.

Ammonia production capacity is split between downstream fertilizer and industrial demand, with the former use accounting for about 75% of production. Industrial or 'technical' demand represents the use of ammonia as a feedstock for production of various industrial chemicals such as caprolactam (for fibre production), acrylonitrile, adipic acid, isocyanates (for polyurethane production), and low density (explosive grade) ammonium nitrate. Caprolactam production consumes 5 million t/a of ammonia, acrylonitrile about 3 million t/a, and hydrogen cyanide production about 500,000 t/a.

Because of higher value end use products, industrial consumers can often pay higher prices for ammonia and so are able to buy ammonia on the merchant market. The same is also true for ammonium phosphate fertilizer producers, which clusters in regions that mine phosphate rock and may not have access to domestic ammonia production. For this reason, technical ammonia and MAP/DAP producers dominate merchant demand for ammonia, although a small amount is imported by urea and ammonium nitrate producers in regions with relatively high domestic gas costs which makes local ammonia production less economic, such as southern Africa.

Geography of supply and demand

Figure 1 shows the location of merchant ammonia production capacity. It is concentrated in relatively low gas cost locations with goods access to ports, and is dominated by Russia, Trinidad and the Middle East. It is also relatively concentrated in terms of suppliers – seven countries were responsible for 80% of all ammonia exports in 2019. Demand, however, is much more diffuse. Figure 2 shows the comparable destinations for merchant ammonia. The top nine importing countries represented only 60% of global ammonia imports in 2019. India, the USA and Morocco all import ammonia for domestic phosphate production, Korea, Belgium and Germany for industrial chemical production.

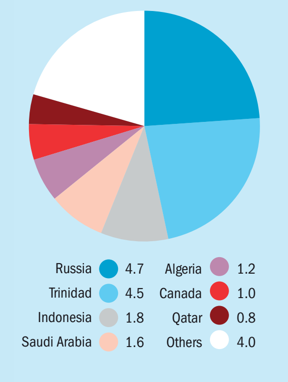
However, the relative importance of these producers and consumers continues to change. The US used to be a much larger importer of ammonia, for example, buying nearly 8 million t/a in 2012, but domestic production has revived as cheap natural gas became available because of the surge in shale gas production. Conversely, Trinidad has struggled with gas supply constraints. Russian exports of ammonia have been rising rapidly in recent years, increasing by 38% just from 2017 to 2019.

Table 1: World ammonia production, consumption and trade, 2019, million t/a (product)

Region	Production	Export	Import	Consumption	Net imports
Western Europe	10.9	1.3	4.4	14.1	3.1
Eastern Europe	4.9	0.2	0.7	5.3	0.5
FSU	25.7	4.7	0.8	21.8	-3.9
North America	21.2	1.4	2.5	22.3	1.1
S/Central America	7.1	4.6	1.6	4.1	-3.0
Africa	9.5	1.9	1.9	9.6	0
Middle East	17.2	3.0	1.3	15.6	-1.7
South Asia	19.6	0	2.8	22.3	2.8
East Asia	64.7	2.1	3.5	66.1	1.4
Oceania	2.0	0.3	0	1.7	-0.3
Total	**182.9**	**19.6**	**19.6**	**182.9**	

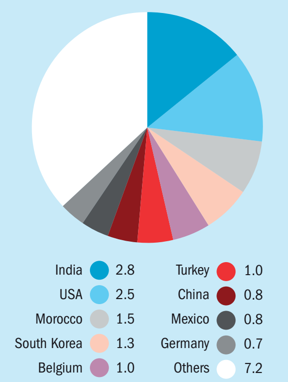
Source: IFA

Fig. 1: Major ammonia exporting countries, 2019, million t/a



Source: IFA

Fig. 2: Major ammonia importing countries, 2019, million t/a



Source: IFA

Trinidad

When gas costs were higher in the US during the 1990s, Trinidad became the favoured location for new ammonia production supplying to the US market as US domestic production in the Gulf Coast closed down. Trinidadian ammonia exports peaked at 5.3 million t/a in 2012. Since then there has been a long slow decline, mainly due to a contraction in US demand as domestic production recovers, and a lack of availability of natural gas. The cost of natural gas has also become a major issue for the island, with the price paid by industrial consumers – mainly ammonia and methanol exporters – set at a lower level than the cost of developing new gas fields. This puts Trinidad's government, which buys gas from local gas producing companies via the Natural Gas Company of Trinidad and Tobago (NGC) and sells it on to downstream producers in a dilemma. If it raises gas prices to the cost of developing new gas fields (a figure of $4.00/MMBtu has been discussed), then it may kill off the ammonia and methanol industries that are the main consumers of that gas. However, if it keeps on supplying gas at the current rate, then the current gas fields will gradually run down. For the past few years NGC has found itself unable to supply 100% of the gas requirements of the various producers on the island and has been rationing gas allocations. This is leading producers to gradually close down production. Nutrien, which operates four 600,000 t/a ammonia plants on the island, idled two of them in May 2020, and has since said that one of them will close permanently. Yara closed one plant in December 2019, and has been operating another line at significantly reduced capacity. Trinidad exported 4.5 million t/a of ammonia in 2019, but the closures and cutbacks have reduced this by about 20% in 2020.

North Africa

The major development in North Africa is the continuing expansion of OCP's phosphate production at its three sites in Morocco. While the timeline of the company's massive pipeline of new MAP/DAP plants has slipped a couple of times, it is still in the middle of a massive expansion. There is not sufficient gas available in Morocco to run the required additional ammonia production, so Moroccan imports have increased, from 0.3 million t/a in 2008 to 1.5 million t/a in 2019. Current proposed projects will require another 1.2 million t/a. Further east, Algeria has been expanding its merchant ammonia exports accordingly, from 0.5 million t/a in 2008 to 1.2 million t/a at present.

Russia

Cheap gas and rising domestic demand for fertilizer is leading to something of a boom for Russian nitrogen production. On the merchant side, EuroChem commissioned a new 850,000 t/a ammonia plant at Kingisepp, near St Petersburg in 2019, with 250,000 t/a of the output earmarked for EuroChem's downstream industrial production in Antwerp, Belgium, as well as domestic ammonium phosphate production, and the company's Lifosa operations in Lithuania. This took Russia's ammonia exports to 4.7 million t/a in 2019, making it the largest exporter of ammonia in the world.

There are several major new downstream urea, urea ammonium sulphate and ammonium nitrate plants under construction in Russia, but a number of them have no associated ammonia production, and will in fact absorb some of Russia's ammonia surplus, leading to lower availability once the plants come on-stream in 2022-23.

Middle East

The Middle East is the third major ammonia exporting region after Russia and Trinidad. Much of its capacity grew on the basis of what was at the time cheap, 'stranded' natural gas in the 1980s and 90s. However, the rapid urbanisation of the region has led to an equally rapid rise in domestic demand for natural gas to generate electricity, and while existing producers, mainly state owned, are still on cheap gas contracts from domestic oil/gas companies, new gas can be quite expensive, especially sour gas developments in Abu Dhabi and Saudi Arabia. Saudi Arabia is also developing its own domestic phosphate industry to try and diversify its economy away from oil. Although Saudi Arabia builds its own gas-based ammonia capacity to supply these MAP/DAP plants, differences in project timing from the start-up of these plants can mean that at times for a year or so Saudi Arabia is exporting large volumes of ammonia.

Elsewhere, Iran exported 1.1 million t/a of ammonia in 2010, but over the past few years has been dogged by US sanctions over its nuclear programme. Iranian ammonia exports fell to 0.3 million t/a in 2019. China has become the main buyer of Iranian ammonia, some of this destined for re-export, but Chinese import requirements for ammonia are relatively modest.

In Oman, some relatively small new merchant ammonia capacity will start up in May 2021 with the new 330,000 t/a Salalah Methanol ammonia plant.

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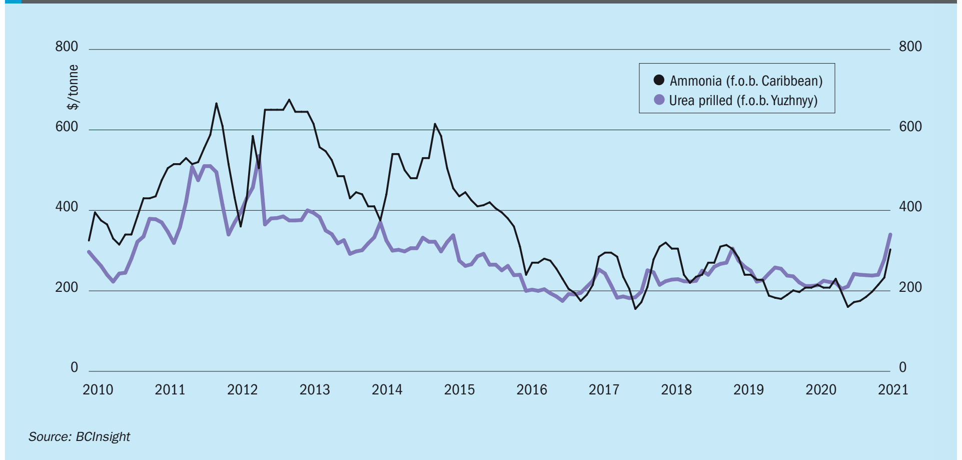
Fig. 3: **Ammonia and urea prices 2010-present**



Source: BCInsight

Covid

The Covid-19 outbreak has naturally had an impact on the ammonia market. In particular, lockdowns in China, Europe and the US affected industrial demand for ammonia in particular. At the same time, very low natural gas prices, especially in Europe, have allowed ammonia producers to keep operating, albeit sometimes at low margins. The result has been continuing weakness in the ammonia market, in spite of intermittent operating problems that have removed merchant capacity from the market. Prices have remained relatively low, as Figure 3 shows.

Global GDP fell by 4.4% in 2020, and this has had a corresponding effect on industrial ammonia demand, although a 5.8% fall in GDP in advanced economies was slightly balanced by a 2% increase in Chinese GDP and 3.3% in developing economies. Conversely, agricultural markets had a good year in 2020, as most governments have treated this as a priority sector and given it considerable support. Crop prices have also been good, which has helped support farmers' buying of fertilizer. Nitrogen fertilizer demand rose 1.7% in 2020. India in particular had a bumper application year, and bought large tonnages of urea. All of this has helped to increase overall ammonia production, as noted earlier, to 190 million t/a. However, it has not significantly impacted the merchant market, as increases in ammonia demand for phosphate production was balanced by reductions in demand for industrial end uses.

Tightening supply

In general the ammonia market remains oversupplied. The merchant market saw a major increase in capacity in 2018-19 and demand, adversely affected by the pandemic is still catching up. However, various factors are likely to see the market tighten over the next few years. In February this year markets had a shock when winter storm Uri descended on the United States, reaching as far south as Texas, and removing power to many fertilizer plants for a couple of weeks. The dislocation in production led to a flurry of US import demand for ammonia, at the same time that several plants were down with production difficulties.

It is likely that as the global vaccination programme continues and covid-related restrictions are gradually eased into the second half of 2021, so industrial demand for ammonia will increase again. At the same time, there are few new merchant ammonia plant additions on the horizon, and some new downstream plants that will remove capacity. The oversupply in the ammonia market is therefore likely to ease over the next couple of years and prices will begin to rise again from the floor that they saw over the past few years.

Looking to the longer term, there are two developments that may begin to impact upon supply and demand further into the decade. Firstly, there is increasing interest in the use of ammonia as a fuel, especially for shipping. In theory, ammonia can be burned to generate only nitrogen and water, and thus is a relatively 'clean' fuel, albeit one that has major challenges relating to storage and handling. The shipping industry is looking towards a potential phase-out of hydrocarbon based fuels towards 2050, and is seriously examining ammonia as a potential candidate for a replacement.

Of course, to be used as a 'clean' fuel, the ammonia would need to be produced using 'green' sources, as we discuss in our article elsewhere in this issue. While at the moment most green ammonia projects are at a pilot or demonstrator scale, Yara has recently announced that it will be progressively switching production at Porsgrunn to electrolysis-based hydrogen, and this is sure to be a growing change in the industry going forward, possibly returning ammonia capacity to places such as Europe where it has atrophied over the past few decades as ammonia moved to where gas was cheap.

This development may also lead to demand for ammonia as a way of storing and transporting hydrogen for a future 'hydrogen economy'. Japan in particular is interested in this development and is working with some renewable ammonia projects in Australia and other places to test the viability of this. For the moment, however, natural gas based production will continue to dominate the industry. ■

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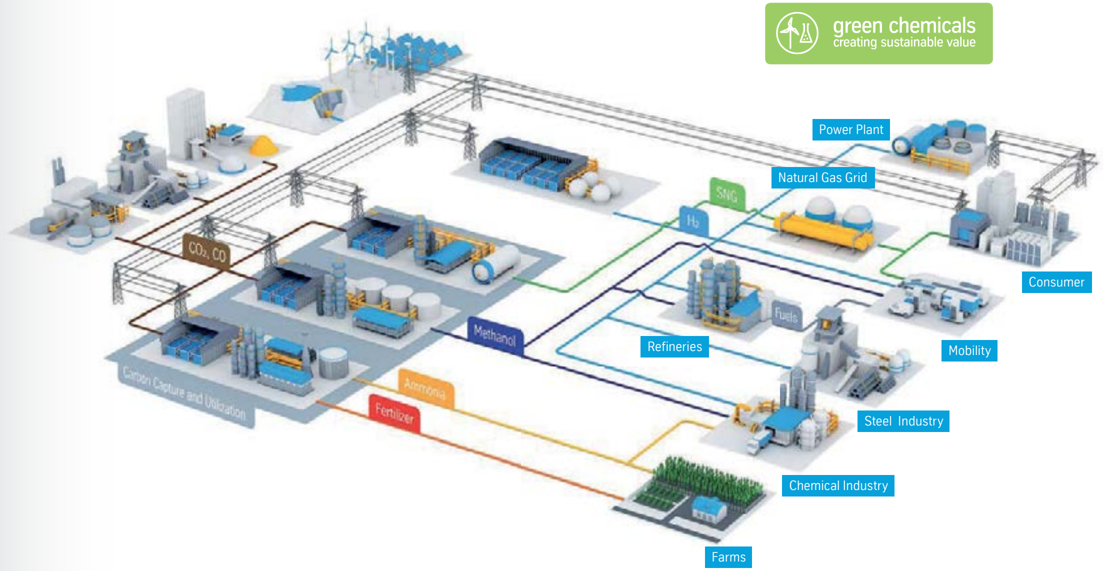

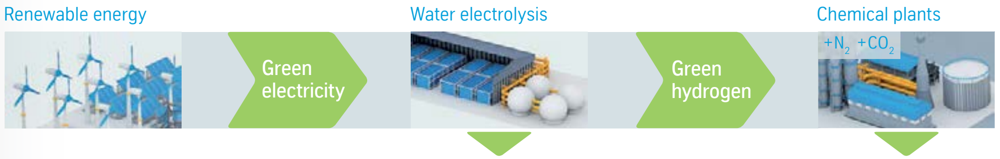

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Towards a sustainable nitrogen fertilizer industry:

Part 2: Decarbonising ammonia production

Georgy Eliseev, Principal Analyst at Fertecon for IHS Markit, looks at the medium to long term outlook for both 'green' and 'blue' ammonia production.

Ammonia production capacity amounts to 220 million tonnes globally, with ammonia production for 2020 being estimated at 190 million tonnes. The vast majority of all ammonia produced, with negligibly small exceptions, is a result of hydrocarbon feedstocks – mostly natural gas or coal – via steam reforming and Haber-Bosch synthesis.

Air provides the nitrogen for the creation of ammonia, which is easily obtained in large quantities from the atmosphere. With minute concentrations in air, obtaining hydrogen is a far more difficult task. At present, the steam reforming of methane is the most convenient way to produce the hydrogen necessary for the synthesis of ammonia in the required quantities and at a reasonable cost. Natural gas, water/steam and air represent the feedstocks, and ammonia, steam and CO_2 are the outputs of this process. In the case of coal being used as a feedstock (mainly in China), a coal gasifier is also required before the reforming stage.

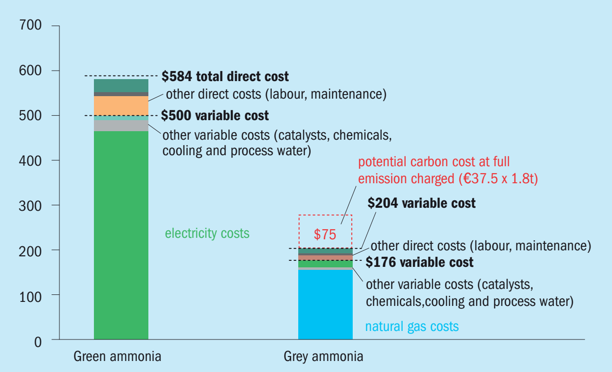
The amount of CO_2 output depends on the rate of natural gas, or energy, consumed in the process. One of the main improvements in ammonia technology since the single train ammonia concept was developed in the 1960s has been in the area of energy efficiency, and the reduction of natural gas consumption per tonne of ammonia. The best plants today consume less than 28 MMBtu of natural gas to produce 1 tonne of ammonia. In such cases, the output of CO_2 will be below 1.6 tonnes per tonne of ammonia. However, the majority of natural gas-fed plants actually consume anywhere from 28 to 41 MMBtu/tonne and so emit from 1.6 to 2.3 tonnes of CO_2 per tonne of ammonia. Coal-based plants emit considerably more than this, approximately 3.5 tonnes of CO_2 or even greater amounts.

With a global ammonia production of 190 million tonnes in 2020, including about 45 million tonnes of coal-based gasification technology, the annual CO_2 emissions from ammonia industry can be estimated at 380-400 million tonnes of CO_2.

Global CO_2 emissions have been estimated at 34-35 billion tonnes of CO2 for 2020, slightly decreasing as a consequence of the Covid-19 crisis. The share of global emissions represented by the ammonia industry is thus estimated at 1.1-1.2%.

More than half of all ammonia produced globally is used for urea production, and the CO_2 captured from the ammonia process is redirected into urea production, as CO_2 is a necessary component for urea. However, once applied to soil urea fertilizer releases CO_2 into the air, and therefore urea fertilizer production is not a good means of reducing carbon emissions. In addition, the use of urea solution for removing NOx in diesel engines and in power plants (AdBlue/DEF) also releases CO_2.

Fig. 1: "Grey" and "green" ammonia direct costs estimates, 2020, $/t



Source: Fertecon

Policy and incentives

Following the Covid outbreak, people are more concerned with the environment and climate change issues in general. Ammonia producers have accordingly come under additional pressure. Shareholders, investors, politicians, regulators, the public; all are intent on seeing immediate plans of action being rolled out to eliminate carbon emissions. Governments are beginning to do away with any support given to CO_2-emitting projects. Hence, accustomed to the relatively low cost of carbon emissions (paying only for emissions that exceed the allowance benchmark), fertilizer companies in many regions can expect costs to rise.

Looking at Europe specifically, the European Emission Trading System (ETS) is one of the most advanced worldwide, and boasts high liquidity among the several regional carbon trade platforms currently in place. A benchmark allowance for emissions based on industry best practices has been set. For ammonia producers, this allowance is set at 1,619 kg of CO_2 for 1,000 kg of ammonia. This corresponds to the average emission of to the 10% most efficient European plants. Producers are not required to pay if they remain under this benchmark, and will pay for any emissions exceeding this allowance. From 2021, the benchmark will be reduced year by year, which will lead to an increase in costs for ammonia producers.

Recently, there has been considerable growth in CO_2 prices in the ETS, figures rising from ~€25 in 2020 to €38 in Feb 2021. However, this level is still not yet sufficient to provide the necessary competitiveness for green ammonia. It is logical to expect that with the growth in demand (as producers will need to buy more carbon allowances at the ETS) carbon prices will also naturally see a rise. At the same time, government and public funding for the support of green ammonia projects will be essential over the next 10-15 years unless green ammonia costs can somehow decrease and become competitive.

Efforts to decarbonise ammonia in Europe (and in other regions) will also inevitably lead to the introduction of border barriers, brought in in order to block 'carbon leakage'. The new Carbon Border Adjustment Mechanism in Europe, taking the form of an import carbon duty or an extension of ETS rules to apply to foreign suppliers, is expected to be in place by 2024.

'Blue' ammonia

'Blue' ammonia differs in only one way from conventional or 'grey' ammonia: the CO_2 flow from combustion is either captured and stored or sequestered; it is not redirected into the urea process. Instead, it is stored or used in such a way which prevents it from escaping into the atmosphere. However, blue ammonia is not exactly carbon-free, as this process has not been perfected and some small quantity of carbon can still make its way into the atmosphere, hence it would be better to call this type of ammonia 'low carbon'. It is considered to be a temporary solution while green ammonia technologies are maturing.

Carbon dioxide from the ammonia process can be injected underground and sequestered in some geological formations. Such projects exist, the Wabash Valley Resources in the USA and Horizon Energi in Norway, for example, as well as a number of projects under consideration in Australia and Canada. Each of these projects will have a long future helping to get rid of carbon by injecting it underground, usually in a liquefied form, however this method cannot be used for all the carbon emitted by the ammonia industry as a permanent answer to the problem.

> **By 2035 the costs of green ammonia may become competitive with those of grey ammonia.**

Another similar way is to take the CO_2 produced and inject it into oil fields in a process known as enhanced oil recovery (EOR), in which gases such as CO_2 are injected into oil layers so as improve the efficiency and yield of oil production by pushing additional oil into a extraction wellbore. This practice has been actively used since the 1970s. The disadvantage of this method is that CO_2 is sequestered, but at the same time more carbon is extracted with the oil taken out.

CO_2 can be converted into solid carbon substances, such as carbon black, graphite, or fly ash. These materials have their applications, although these are somewhat limited. Saudi Aramco uses this concept to produce polypropylene carbonate polyols with CO_2 instead of methane as a feedstock. Other ideas also exist with regard to eliminating or soaking up the CO_2 created during the production of ammonia, such as producing chemicals, plastics, nanofibers and so on. CO_2 can also be used to improve the quality of concrete. Finally, there is the biological sequestration of CO_2 by plants into soil. Scientists are working on ways to accelerate this process. This may represent a long-term solution for CO_2 stream sequestration; making artificial soil and using it to soak up carbon.

'Green' ammonia

For full decarbonisation a carbon-free way for the production of ammonia is necessary, and this is the idea of green ammonia – which is based on the electrolysis of water. Some 10.5 MWh of green electricity are needed to split a molecule of water and separate hydrogen from oxygen. Oxygen is a by-product from this process. Unlike in the steam-reforming process where air is injected to add nitrogen, in the green ammonia process nitrogen should be separately produced in an air separation unit. Then, both hydrogen and nitrogen are directed into the same Haber-Bosch synthesis to be converted into ammonia. In this process, carbon is not present in any sense, so this really is a carbon-free process.

The technology of green ammonia is under development at the present time, and it is already known that the energy consumed by electrolysis may be reduced to less than 8 MWh. This is vital for the use of ammonia as an energy carrier, as its energy content is 5.2 MWh – so the loss of energy during this transformation to ammonia is currently about half. Moreover, new catalysts may be developed to reduce temperature and pressure in the ammonia converter, which should reduce costs accordingly. There is an idea that one-stage green ammonia production could be viable, so that not hydrogen, but ammonia rather would be produced during electrolysis.

Costs gap

There is still a notable gap in both the Opex and Capex for green and grey ammonia. The direct costs, including energy, chemicals, catalysts, process and cooling water, labour and maintenance, calculated for Europe, are almost 3 times higher for green ammonia (Figure 1) than for traditional ammonia. Even if the full costs of carbon emission credits were added to the grey ammonia costs, green ammonia will still remain uncompetitive. Electricity represents about 80% of the direct costs of green ammonia, whereas natural gas represents 76% of grey ammonia costs.

Reducing the costs of green electricity along with the development of green energy (via economies of scale) and advances in technology, on the one hand, and the introduction of carbon taxes and rising natural gas prices on the other, will reduce the Opex gap, so that by 2035 the costs of

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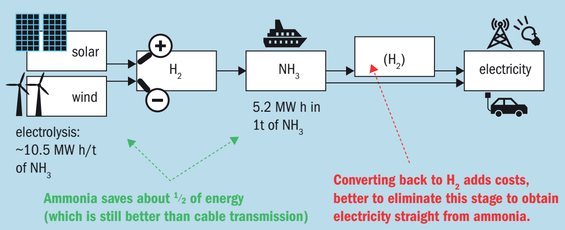
Fig. 2: **Ammonia as an energy carrier**



Source: Fertecon

green ammonia may become competitive compared with those of grey ammonia.

Regarding the Capex, our estimates show that the capital expenditure per tonne of nominal ammonia capacity amounts to $2,700 for a hypothetical green ammonia plant (400 t/d), or $1,500 in the case of a small green hydrogen plant being added to an existing ammonia unit. The Capex of recent standalone grey ammonia projects was from $826/t (Yara/BASF Freeport 2018 – a short cycle plant which receives H_2 and N_2 from a supplier) to $1,203/t (PAU Indonesia 2018, full cycle), and up to $1,342/t (Salalah Oman 2021, a smaller sized plant).

Is there enough green energy?

However, is there enough green energy to decarbonize the ammonia industry as it is at present? If the electricity requirement of electrolysis units if all existing 'grey' ammonia facilities were replaced with green is compared with the current output of solar, wind and biomass electricity available in 2019, it can be seen that total decarbonising of ammonia production would require 67% of currently existing green energy, and – in many regions – more than 100% of existing green energy capacity. If one decides to invest in a green ammonia plant, the first question is: where can this green energy be obtained from? And in most cases the answer would be to invest first in a solar or wind power plant.

The future of urea

One additional point which could slow down the process of decarbonizing the fertilizer industry is urea. Urea is the most popular nitrogen fertilizer across the world, and global consumption in 2020 was over 176 million t/a (China alone consumes 50 million t/a, and India 34.4 million t/a). Urea production absorbs more than 50% of ammonia produced globally. In the absence of a source of CO_2 (in the case of green/blue ammonia), urea will not be produced. However, farmers in India, China and a number of other countries are used to urea, and it will be rather difficult to find a substitute.

Green urea may be produced using biomass, according whether biomass is available in large quantities, while the utilisation of CO_2 captured from the air is still extremely expensive, as a consequence of the very low concentration of CO_2 in air – only 0.04%.

Strategies for fertilizer companies

At present, there are several green ammonia projects at various stages of implementation in the fertilizer industry. These projects may be divided into 3 groups.

- The first group consists of only one plant, which is scheduled to be completely electrified in the future. The Yara Porsgrunn plant produces 0.5 million t/a of ammonia and is located in Norway. The company has stated that provided power is available at the site and the required public co-funding is in place, the project could be realized within 5-7 years. In fact, electrolysis for this plant is likely to consume 38% of Norway's wind power. Norway does also possesses a large amount of hydro-electric power generation, so this may also be used for decarbonising Porsgrunn.
- The second group is represented by a number of projects, including Yara Sluiskil in the Netherlands, Yara Pilbara in Australia, Fertiberia in Spain, and CF Industries in the US, which plan to start working with low levels of electrolysis, which will provide their existing ammonia converters with some percentage of carbon-free ammonia, usually about 10% of overall ammonia production. There will be no increase in ammonia capacity, but a there will be a reduction in natural gas use which will be, in turn, replaced by green hydrogen. The average emission per tonne of ammonia will thus be reduced by 10%. The first project from this group may be already on stream this year in Puertollano, Spain, where Iberdrola will supply hydrogen to the Fertiberia ammonia plant.
- The third group consists of fertilizer and industrial nitrate producers who import ammonia, and green ammonia plants will allow companies to reduce or eliminate imports. OCP in Morocco and Enaex in Chile, with partners, are considering such plants. Both countries are blessed with sufficient sunshine to provide green ammonia projects with solar power.

Oddly enough, though, as yet there are still no blue ammonia projects in the industry, which could be seen as a more affordable alternative.

Carbon-free ammonia for green power

Ammonia contains hydrogen and is easier in terms of handling than hydrogen itself. Therefore, it is logical to use ammonia as an auxiliary element in hydrogen energy economics, for example, for the transportation and storage of ammonia.

There are places on the Earth with continuous, long-lasting, and predictable sunshine. This, combined with wind power to cover periods of darkness, mean that hydrogen and ammonia can be produced with very good economics. Investments in green energy will be directed into such areas, and green energy should then be transported to the areas where it will be used. This will lead to the emergence of green energy trade flows between the continents).

Green ammonia may also be the best form in which green hydrogen (or green energy) can be shipped from e.g., Australia to Japan or Europe, or from North Africa to North America and so on. Although converting electricity to ammonia leads to the loss of half of the energy which could be yielded, it is still better than the losses involved in cable transmission over long distances, and one of the few ways to facilitate energy seaborne transportation (Figure 2). There are still additional methods of reducing costs and improving the efficiency of ammonia for the transportation of energy.

Ammonia can also be used as a means of energy storage, given that green energy is highly intermittent and often not easily transmittable, while the electricity produced is cyclical with short term oscillations between peaks and off peaks. From the perspective of both supply and demand there is a need for the storage of energy, and ammonia may be used for this purpose.

Ammonia can be also used as a fuel for certain applications, thus being able to compete with hydrogen. It has certain advantages to hydrogen as a fuel for maritime transportation. The International Maritime Organisation has set rigid targets to reduce the carbon footprint of shipping, which accounts for 2% of global emissions, by at least 50% by 2050 compared to 2008. These targets can be only achieved if, along with low-carbon fuels (such as LNG or methanol) some zero carbon fuel is used. At the present time, hydrogen and ammonia, although containing less energy per tonne, and requiring much more space in bunker tanks than conventional maritime fuel, potentially can be used for this purpose. If 20-25% of maritime fuel was replaced by ammonia right now (not even considering any future growth in traffic), it would require more green ammonia than the existing capacity of grey ammonia.

There are several interesting initiatives for green and blue ammonia production for energy purposes. NEOM is an economic zone located in northwest Saudi Arabia. A $5 billion project with a capacity of 1.2 million t/a of green ammonia is scheduled for 2025. Air Products, a partner in the project, will take the green ammonia from the NEOM site and intend to transport it around the world to be converted back into hydrogen at local hydrogen refuelling stations.

Copenhagen Infrastructure Partners (CIP) have announced a project in Esbjerg, Denmark, to convert power from offshore wind turbines into green ammonia. Capacity is estimated at over 800,000 t/a of green ammonia. BP Australia is planning to start with a pilot project in Geraldton, West Australia, with a capacity of 20,000 t/a and a possible expansion of up to 1 million t/a. A number of other green ammonia projects are planned in Europe and Australia.

Wabash Valley resources will be the largest blue ammonia project globally and will have a capacity of 550,000 t/a and is planned to open in 2022 in the USA. Additionally, there are also a number of potential blue ammonia projects planned for Europe (Horizon Energi), Australia and Canada.

Forecast

Although we do not have sufficient data at present, and there is a large degree of uncertainty, we forecast that carbon-free ammonia production may rise from virtually zero in 2020 to 5 million t/a by 2030, and to 20-30 million t/a by 2035 (Figure 2). If the costs of green ammonia become competitive by the middle of the 2030s, then it is probable that a more rapid expansion of green ammonia will take place, with green replacing grey ammonia after 2035.

The use of ammonia as a fuel or as a hydrogen carrier for power may create a very large industry in the future, with production volumes much exceeding the existing ammonia industry. ■

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CRU Nitrogen + Syngas 2021

Because of the ongoing pandemic, this year's CRU Nitrogen + Syngas conference was held as a 'virtual' event, in early March 2021.

CRU's annual Nitrogen + Syngas conference is one of the annual fixtures on the calendar for those industries, and while covid continues to make travel impossible, this year's virtual event provided a good mix of interesting papers with question and answer sessions and interaction with virtual exhibitors and attendees. Some of the highlights follow.

Markets

As usual, the conference kicked off with the nitrogen industry overview, this time presented by CRU's Alexander Derricott. Both ammonia and urea have weathered the covid storm relatively well. Urea demand was up around 5.7 million t/a during 2020, with notable increases in India, China and Australia and production increases seen in Russia, China and Indonesia, offset by falls in Malaysia and Europe. Fertilizer demand has held up well, as governments decided agriculture was a key strategic resource, though technical demand fell. Urea trade reached 51.4 million t/a, but prices have remained weak, continuing the low level they have maintained since 2016, with new capacity generating oversupply. The collapse in feedstock prices seen in 2020 have also kept price floors low, although there has been a spike in LNG prices in early 2021. Moving into 2021, crop prices have jumped, with Chinese soy and corn demand high to rebuild swine herds decimated by disease. Good crop prices have encouraged farmers to buy more fertilizer. A wave of new urea capacity in 2021-22 (albeit some delayed due to covid) will probably keep a lid on prices for this year at least, but looking further ahead, the demand outlook is robust for both ammonia and urea, with total nitrogen demand rising to 167 million tN/a by 2025, and this is forecast to roughly balance new capacity rises, depending upon the rate of Chinese capacity closures. A rebound in energy prices will see costs rise, even in some countries that currently fix gas costs for fertilizer producers, and as utilisation rates rise so prices should also rise.

Looking to the longer term, Alexander said that the nitrogen industry has a CO_2 challenge. There is an increasing focus by companies and governments on environmental, social, and corporate governance (ESG) issues. There is a major emissions difference between gas and coal-based production – the latter emits much more CO_2. There are a number of options for decarbonising ammonia, which are discussed in much greater detail below and elsewhere in this issue.

Alexander was followed by his CRU colleague Arham Muhammad, who looked upstream at natural gas pricing – a key determinant of ammonia and urea pricing. The cold weather this winter, especially in the US, have contributed to a major LNG price spike. January's reduction in output came together with supply outages in Australia, Malaysia, Qatar, Norway, Nigeria, and Trinidad. Most countries secure 70% of their LNG requirements on long-term contracts - this means that when the market is tight, spot volumes may not be available quickly and prices can spike rapidly. The LNG market has been oversupplied for a few years, exacerbated by demand falls early in the covid pandemic. However, there are now signs that we are moving into a market where net demand growth is outpacing new capacity additions.

Low carbon ammonia

For the rest of Monday, several papers presented the views of major licensor companies about low carbon ammonia production. Klemens Wawrzinek of Linde began with a look at 'blue' hydrogen production. Hydrogen will play a key role in the transition to cleaner energy, with 2018 demand of 118 million t/a likely to double by 2030 and multiply tenfold by 2050. The EU's CertiHy certification scheme defines blue and green hydrogen as anything with less than 36.4g of CO_2 emissions per MJ of hydrogen (LHV) – or about a 40% reduction in carbon intensity of production compared to conventional 'grey' hydrogen. It is unlikely that sufficient electrolysis capacity can be built to satisfy projected H_2 demand. There are therefore a number of 'blue' pilot carbon capture projects around the North Sea using depleted oil and gas reservoirs to house CO_2. Klemens presented a case study using several flowsheets – conventional (both reforming and partial oxidation), partial carbon capture and full carbon capture, and an electrolysis comparison case. Even generating hydrogen from green electricity has some CO_2 emissions, and the figures showed that these were comparable with a reformer using full, flue-gas CO_2 capture, while the latter had only 60% of the cost per tonne of CO_2 emissions avoided, and the lowest unit production cost of H_2 for any low carbon option, unless power is available at very cheap cost.

Michael Reinke of Linde moved on to green ammonia production. Linde has joined with PEM manufacturer ITM Power in ITM Linde Electrolysis to produce modular electrolysers. At present modules are 2MW, but a 5MW module will be available from 2023. It has also developed FLEX-ASU, an air separation unit optimised for cyclic operation that can vary its output from 30-100% production for use with electrolysis based ammonia production. Various buffer technologies can also be used to smooth output – electric grid export, battery storage, and ammonia or hydrogen storage either as a liquid or gas.

Klaus Noelker of thyssenkrupp Industrial Solutions noted that use of ammonia could also double by 2050 if it becomes widely adopted as a marine fuel. He focused on 'green' ammonia, via electrolysis. The challenge is to match fluctuating power input with the requirement for stable chemical production, without over-specifying equipment sizes. TKIS has a modelling tool called RHAMFS which can specify the optimum sizing of the synloop and hydrogen storage buffer for any combination of circumstances. Green ammonia plants also have less steam from waste heat available to drive syngas compressors. Smaller plants can reduce capex by using waste heat for absorption refrigeration, and driving compressors using electric motors, while larger scale electrolysis plants can reduce opex by feeding the steam to a generator turbine. Existing plants can be retrofitted with additional green hydrogen side streams, but steam balance issues must once again be considered. Cost-wise, the key determinant of the viability of the plant is the cost of green electricity supply. A cost of \$15/MWh or less is needed to make the plant cost competitive, although a \$30/tCO_2e carbon price on CO_2 emissions would raise that threshold to \$20/MWh.

Yawar Abbas Naqvi presented Haldor Topsoe's approach to a lower carbon economy. The company has set itself the strategic goal of being recognised as the global leader in carbon emission reduction technologies by 2024. Topsoe sees hybrid plants where only part of the feed is replaced by electrolysis as a bridge towards full decarbonisation. Our world also requires carbon chemicals for plastics and other materials. Green methanol can be a low carbon carbon source, converted downstream into DME, gasoline, polyolefins etc.

Francisco Baratto showcased Casale;s green and blue hydrogen and ammonia technologies, though he also added 'red' ammonia (hydrogen electrolysed using nuclear electricity) and 'turquoise' ammonia to the colour palette, where pyrolysis is used to generate ammonia and solid carbon that can be sequestered. Casale have developed its A6000CC process for blue ammonia applications, using pre-combustion carbon capture to reduce the energy penalty while achieving 90% capture. It is an autothermal reformer based process. Hydrogen rich process gas is used to generate heat for the CO_2 capture section. One such plant is under construction in Russia using an MHI CO_2 removal license. It can also be used as a revamp for an existing plant, or adapted for blue hydrogen production. On the green ammonia side, Casale's A60 and A600 processes are geared towards green ammonia production, designed for smaller scale highly automated production. System optimisation uses a software tool to reduce the cut-off of renewable power (i.e. when hydrogen storage is too full or when generation is too low and backup power must be used). Having a flexible synloop that can work from 10-110% capacity is the key to the system, and lowers ammonia production cost by 15%. Once again, green hydrogen can be used in hybrid plants.

Technology

Some of the technology showcased at the conference included the *ActiSafe* catalyst handling system, described on p48, Navigance's data-driven analytical system, described on pp54-57, and some of the efficiency improvements available for urea plant operators that can be found in our article on pages 28-40 this issue.

Martin Østberg of Haldor Topsoe introduced a novel CO_2 reforming technology for production of CO-rich synthesis gas at a low steam:carbon ratio, designed for use in conjunction with carbon capture technologies.

OMNI Conversion Technologies has developed the Plasco gasification and plasma refining system (GPRS) for the production of clean chemicals from waste. Martin Bacon described the process, and said that OMNI has now developed a 200 t/d modular gasification system with high reliablility (92% availability) which can be installed in parallel streams for scale-up.

On the environmental side, René Braun of Grandperspective GmbH described the installation of an early warning solution for gas leaks at OCI's CHEMELOT facility in the Netherlands, while Andrea Carotti of Saipem presented a new electrochemical wastewater treatment technology for emissions abatement. BD Energy Systems described the successful installation of a selective non-catalytic reduction (SNCR) system for NOx emissions reduction in a steam reformer. Alessandro Gullà of AWS Corporation presented an electrostatic precipitation system for dust control in AN/CAN plants.

Catalyst presentations covered the first commercial references of Clariant's *ReforMax*® LDP Plus series and associated reformer service *ReforSafE*™; operating *ShiftMax* reforming catalyst under harsh conditions; computational fluid dynamic modelling by TKIS and Umicore to improve ammonia oxidation catalyst design; Johnson Matthey looked at the impact of catalyst size, shape and strength on performance, and the decisions that had led to the development of its F-series catalysts.

Operator experiences included a case study from PT Kaltim Parna Industri in Indonesia on incident management of natural gas pipe leaks; the structural rehabilitation of a wooden cooling tower at Engro Fertilizers Pakistan; life extension of two bimetallic strippers by Iffco in India; and improving energy efficiency and resolving a performance decline in a KBR Purifier-based plant by PT Petrokimia Gresik. ■


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Maximising urea plant efficiency and reliability

From new materials of construction and improved equipment designs to the latest digital tools, Casale, thyssenkrupp Industrial Solutions, Saipem, TOYO and Stamicarbon report on some of their latest achievements.

The reliability of a system, in the general sense of its meaning, is the ability of achieving its design scope within the expected lifetime, ensuring product quality and process availability by operating in compliance with the key health, safety and environmental requirements.

Nowadays, more than ever, the process industry is looking at the concept of reliability from a different perspective. It applies to the design, engineering and operation of new grass roots units and especially to the revamping of older industrial plants.

A major percentage of the world's urea production comes from old installations, which were designed and built decades ago. Today it is not only the ageing effects that threaten production from these vintage plants, market conditions are challenging and environmental and safety regulations are more stringent than ever.

Whereas in the past it was common to see mechanical engineers involved in maintenance activities, today organisations have institutionalised groups of competence, still including the function of the mechanical engineer, but working jointly with the process, electrical and instrumentation and automation engineers.

In a modern concept a reliable urea process should be operationally ready, safe and efficient at all the times. To guarantee financial success and market competitiveness, urea production should be predictable in terms of throughput and product quality and the process should be capable of achieving the highest performance with the minimum operational expenditure. In general, the process should also comply with the newest environmental and safety regulations and be designed to meet efficiency targets.

The introduction of the various stripping processes for urea production started in the 1970s and was a major advancement for the efficiency of urea production. Both CO_2 and thermal stripping processes reduced the energy intensity by almost 50% compared to traditional total recycle processes. However, this breakthrough in process technology also brought major challenges in terms of plant reliability.

In modern urea plants, the decomposition and recycle of unreacted carbamate at high pressure and temperature occurs at operating conditions much harsher than those typical of traditional processes, demanding superior materials for higher durability as well as particular care in process operation and maintenance activities to achieve and sustain the highest safety and reliability standards throughout the entire life of the plants.

Historically, alloys such as titanium and zirconium have been widely employed because of their excellent corrosion resistance properties. Later, super austenitic urea grade steel such as AISI 316L UG and 25Cr.22Ni.2Mo were widely used for the design of critical equipment and plant components with the aim of reducing the impact of the material on the cost and to further improve reliability.

Today, super duplex materials are widely used to improve reliability and safety, as well as providing significant advanages in terms of process design and efficiency, in grass roots, revamping and major capital equipment replacement projects.

CASALE

Uremium29 brings a leap forward in equipment reliabilty

M. Fumagalli, L. Rugnone and P. Bertini.

Casale, as technology licensor, brings added value to any project for revamping or grass root urea production plants thanks to its competence developed over several decades and to its capability to bring together technology and mechanical engineering competence in a single project. Casale has improved and consolidated process schemes, materials of construction and the mechanical design approach to drive down the consumption of medium pressure steam in urea plants.

Production plants can also benefit from Casale's valuable experience in process operation through its wide range of digital services aimed at remote monitoring and optimisation.

A leap forward in equipment reliability has been brought about by the introduction of new super duplex steel. Super duplex materials are characterised by a higher content of chromium and lower content of nickel compared to the super austenitic steels previously employed. Such chemistry allows for ferritic and austenitic layers to be bonded into a "sandwich" structure exhibiting superior corrosion resistance properties.

In joint cooperation with Tubacex, Casale has developed and extensively tested Uremium29, a super duplex material for HP urea service.

The introduction of Uremium29 and the latest approach to equipment design allows not only new grass roots installations, but also vintage small-to-medium urea plants to remain competitive after decades of service. The goal of most owners of vintage urea plant is to prolong the lifetime of their facilities with focused maintenance interventions, end-of-life equipment replacement and at the same time small- to medium-scale debottleneck revamping to achieve opex figures in line with the most modern grass roots installations.

In this context it is understood that the industrial urea community is looking at reliability from a different perspective, i.e. plant performance should be supported by both mechanical and process reliability, otherwise an efficient, but costly, modern stripping plant can experience a more drastic erosion of its gross margin for any unexpected mechanical failure, prolonged downtime or slowdown of production than a vintage less efficient plant.



Left to right: HP Uremium29 stripper, Uremium29 components in the HP stripper top and Uremium29 tube to tubesheet orbital welding.

PHOTOS: CASALE

Uremium29 – reliable efficiency

Uremium29 is an austenitic-ferritic steel with high chromium and low nickel contents in accordance with requirements of ASTM UNS S32906 and ASME Code Case 2295.

Since its first delivery to the market, in 2016, Casale has accrued a number of Uremium29 orders for new critical HP equipment. The material reliability and suitability was broadly tested for an extensive range of urea process conditions in strict lab tests and in plants before being released to the market. Casale was determined to exploit its superior mechanical and chemical properties to boost the life expectancy of critical urea equipment without compromising on plant efficiency. Uremium29 find its natural application in all parts in contact with process fluids of the synthesis section of stripping plants. In addition to critical equipment, it is also highly suitable for piping, fittings, control valves and critical components of process instrumentation.

Besides these virtues, the enhanced repassivation capacity of Uremium29 has an immediate impact on the efficiency of urea synthesis without compromising on plant reliability. In CO_2 stripping plants for example, where parts in contact with the process fluids of the HP stripper are made of traditional 310 MoLN steel, the required oxygen fraction in the CO_2 feed ranges from 0.6 to 0.8 vol-% which has a detrimental effect on conversion in the urea reactor. Use of super-duplex such as Uremium29 allows a drastic reduction of oxygen for passivation, down to 0.3 vol-%, which typically improves conversion with consequent reduction of MP steam consumption by 2.0 to 3.0%. Additionally, the increased resistance to corrosion, and the superior mechanical strength, allows thinner tubes to be used. Thinner tubes reduce the overall weight of the item which becomes extremely useful when replacing existing vintage strippers as it makes it possible to increase the number of tubes without the need for reinforcement of the support structure and foundations. Tubes with larger internal diameters handle an increased maximum load per tube prior onset of flooding. In existing vintage urea plants where the HP stripper is frequently the capacity bottleneck, simply replacing the existing item with modern strippers equipped with Uremium29 tubes often creates enough room for a moderate plant capacity increase. This concept also applies to other critical items in urea stripping plants; for example, replacing a traditional 310 MoLN HP carbamate condenser with a new one equipped with Uremium29 thinner tubes allows for a larger surface area at comparable equipment weight. The new HP carbamate condenser would generate LP steam at higher pressure than the replaced item, therefore opening up the possibility to debottleneck downstream equipment and increase plant throughput.

Uremium29 is also ideally suited for application in process locations where the presence of two-phase liquid-gas flow and high fluid velocity may activate erosion induced corrosion phenomena. It is a useful choice for any equipment internal, two-phase flow piping and especially for the design of the synthesis letdown valves, which need to withstand heavy duty service for many years.

The use of Uremium29 as material of construction for HP strippers is not limited to CO_2 stripping; Casale also applies Uremium29 to self-stripping technology which traditionally relies on zirconium, titanium or bimetallic (25Cr.22Ni.2Mo + zirconium) tubes to withstand the high temperature at the stripper bottom. In the most typical process embodiment where a bimetallic stripper is employed, air compressors are provided to satisfy the passivation needs and protect the stainless steel internals, liners and weld overlays against corrosion. The most critical location is actually the stripper outlet, where the average working temperature reaches 204-205°C.

Casale's philosophy for self-stripping technology foresees fully exploiting the potential of Uremium29 by replacing the bimetallic equipment used in combination with the improved NH_3 stripping process configuration. This solution consists of installing a level in the reactor top, typically by replacing the reactor cover. In this way, the vapour phase at the reactor outlet, which still contains oxygen, is separated from the liquid and is driven by a new line up to the new Uremium29 stripper bottom to provide oxygen for passivation purposes. In this way, passivation air compressors, which are highly demanding in terms of maintenance and are typically a source for oil entrainment into the process, can be idled. The overall pressure of inerts in the synthesis loop and ammonia recovery sections of the urea plant is also reduced providing a great benefit in terms of condensation and ammonia capture capacity.

Casale operational assistance

The operation of a urea process is a very complex task. The complexity arises from the intricate interaction between participant chemicals and from the need to manipulate a large set of variables to reach production targets, while maintaining optimum efficiency in terms of consumption and emission figures.

The close relationship between process operation and mechanical reliability actually adds an additional element to the complex operation of chemical plants. Often, plants are highly stressed by the operating conditions which are pushed to the limit to maintain profits. This is particularly true

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for old plants whose mechanical reliability and related performance must be carefully monitored. Therefore, it is of extreme importance to develop maintenance programs aimed at improving reliability. The operational data becomes a fundamental part of a more extensive analysis that not only supports the diagnostics of failures but also helps to understand the root causes during mechanical inspections.

To help plant operations to achieve and maintain the best efficiency, support maintenance and further improve reliability, Casale has created a portfolio of digital products that take full advantage of the existing plant data infrastructure:

- Casale Remote Engineering Service (CARES), providing valuable support aimed at achieving the customer's production targets, improving process reliability and supporting mechanical inspections;
- Casale Operator Training System (OTS), to improve the confidence and knowledge of operational teams, because people are the most valuable asset, the internal factor in driving the plant close to the optimum efficiency;
- Casale Model Predictive Control (MPC), a trusted "auto-pilot", embedding process knowledge to keep the plant cruising at its best efficiency, every moment, every day;
- Casale smart instrumentation providing reliable tools to assist operations: Advanced URea Online Raman Analyser (or AURORA), tunable diode laser, refractive index meter, HP urea service guided wave radar level, etc. ■

THYSSENKRUPP INDUSTRIAL SOLUTIONS

Higher operating stability with a self-regulating pump

C. Will and M. Wieschalla

One of the well-known challenges in operating a urea plant is to keep the biuret formation to a minimum in order to avoid off-specification product. Biuret formation is an unwanted but inevitable side reaction of two urea molecules polymerising with elimination of ammonia. The formation is accelerated by three factors: low ammonia concentrations, high temperatures and high residence times. There are a few locations in every urea plant, where all these factors occur at the same time. In this article the focus is on the outlet of the evaporation section and the melt transfer line to the granulation unit.

In order to transfer the urea melt leaving the evaporation section to the granulation unit and to ensure the required supply pressure for the granulator spray nozzles, a dedicated pump is required.

Considering that the evaporation section is operated at vacuum conditions and the melt leaving the evaporator is boiling, the NPSH (net positive suction head) for that transfer pump has to be accordingly high. A common value is 3 m. But having a suction head of approximately 3 m in order to avoid cavitation, consequently leads to a higher residence time of the urea melt in the pump suction line and thus to increased biuret formation.

To avoid this scenario, thyssenkrupp Industrial Solutions (tkIS) optionally equips newly built plants with a special self-regulating pump. Compared to a conventional centrifugal pump, the self-regulating pump is independent of the intake rate, i.e. the pump capacity always corresponds to the intake rate resulting in a required NPSH of approximately 0 m. Consequently, the required suction head and therefore the residence time of the melt are decreased to a minimum, leading to less biuret formation and avoiding plant trips due to a loss of level.

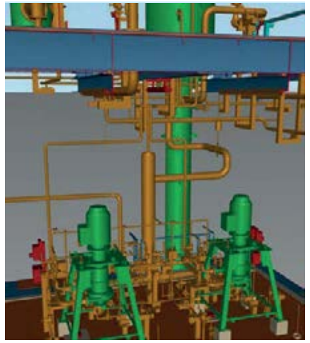


Fig. 1: Typical setup of a self-regulating pump.

A fertilizer complex consisting of an ammonia and a urea plant normally runs smoothly without significant capacity changes. But, in case the complex consists of more units, e.g. urea synthesis, urea granulation, diesel exhaust fluid unit, UAN, which the urea plant has to supply with urea solution or melt, the evaporation unit of the urea synthesis and the granulation plant can suffer from an unstable supply of urea solution and melt. In these cases, the operator has to adapt the load to the evaporation section and consequently the transfer pump capacity accordingly. Especially when the evaporation feed has to be reduced, care has to be taken in cases where a conventional centrifugal pump is used. Reducing the evaporation feed can easily initiate the dry run protection of the pump in the suction line, resulting in tripping of the downstream granulation unit. However, if a self-regulating pump is used, that scenario will not happen. Due to the self-regulating character of the pump, the capacity will adapt according to the intake rate without causing a trip of the pump and the downstream process unit. Thus, capacity fluctuations can be easily handled by this type of pump, facilitating the plant operator's work and reducing the risk of tripping downstream units.

Another advantage of the self-regulating pump is the elimination of layout constraints for the outlet of the evaporation section. When using a conventional centrifugal pump, a required suction head has to be maintained. Depending on the plant layout, the line between the outlet of the evaporator and the suction flange of the pump can be quite long, increasing the melt's residence time and thus promoting biuret formation. A self-regulating pump can be located directly below the evaporator outlet, since no specific suction head is required. Only the overall height of the pump frame and the required head space has to be considered. Fig. 1 shows a typical pump setup. It is clearly visible how close the pump can be placed to the evaporator, which can be seen in the background.

Over the past years tkIS has gained a lot of good experience using the self-regulating pump with respect to efficiency and reliability. Furthermore, the plant layout has improved specifically at this challenging location. The self-regulating pump provides security for a stable operation, especially for the downstream granulation unit. tkIS holds the patent rights to install a self-regulating pump at this location. The self-regulating pump can be installed for grass-roots plants as well as during revamping. ■

SAIPEM

A proven approach to achieving better equipment life

L. E. Viganò and F. Mussa

While good design is the starting point for long-lasting equipment, it is not the sole answer to the problem of ensuring reliability of urea plants, according to the experience of Saipem, as licensor and EPC contractor of the Snamprogetti™ Urea Technology. Care during the construction and throughout the whole lifetime of the equipment is also essential.

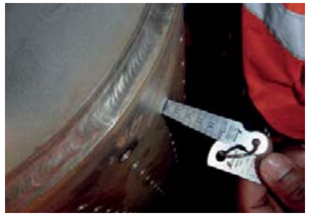
Saipem's main focus of its proprietary urea technology centres on the high pressure equipment due to the criticality of their operating and design conditions.

Saipem draws upon its experience in plant and equipment design spanning a wide spectrum of fields, ranging from green technologies to the oil and gas industry, to find the most appropriate solutions for the optimisation and improvement of equipment design.

By establishing close and enduring relationships with manufacturers, equipment can be designed with the knowledge of any associated construction issues, ensuring that the proposed solutions are technically viable, thus avoiding unexpected, expensive and time-consuming reworks aimed at finding alternative solutions which, in the rush to meet the project schedule, may not be the optimal ones.

It is these experiences and bonds with manufacturers that are the basis for the continuous development of the equipment design, as well as, for the definition of criteria, the renowned confidential and proprietary Saipem's "510 & 516" series of specifications. These criteria consist of a collection of requirements regarding materials, methods of construction, examinations and inspections during manufacturing that are periodically updated based on the feedback from manufacturers and operating plants to ensure and to improve the quality of the equipment. The presence of Saipem's quality inspectors during the main steps of construction of its equipment, either in the manufacturer's workshop or, in case of constraints (as experienced during the recent pandemic) remotely, ensures that all fabrication steps are monitored, providing the smooth and timely manufacture of equipment according to Saipem's design requirements and criteria.

Once equipment is installed, Saipem is proud to assist end-users by offering its post-sales assistance, which covers a



Inspection of equipment internals.

HP tubesheet.

PHOTOS: SAIPEM

wide range of services with the target of monitoring the status of the equipment, ensuring (if not prolonging) its expected life time and boosting its performance. In this way Saipem has acquired and developed a deep knowhow for all the different pieces of equipment, specialising in performing the inspections, understanding the best approaches to attend the required maintenance and, last but not least, getting a feeling on how to intervene if features of equipment design require improvement.

A demonstration of the benefits of this approach is the development of the design of the ferrules of the urea stripper. Ferrules are the distribution devices for the urea solution, installed over the head of the tubes, that generate a falling film flow along the tubes of the heat exchangers (in this case the urea stripper) thus providing optimal heat transfer and efficient decomposition of the carbamate and consequent release of the vapours.

By performing the inspections on different plants and relevant strippers, it has been noticed that the portion of the bimetallic tubes heads from where the zirconium was removed was more prone, long term, to corrosion issues; corrosion was due to 25/22/2 Cr/Ni/Mo material (in proximity of the weld) being directly exposed to the process fluid and to the higher temperatures of the tube-sheet.

After a joint discussion among process and mechanical engineers and quality experts, the design of the head of the tubes was modified by increasing the length of the protrusion of the tube ends over the tube-sheet to allow for maintaining the terminal zirconium and consequently avoiding, over long periods, the risk of corrosion on 25/22/2 Cr/Ni/Mo tubes. The proposed design was then presented to the stripper qualified manufacturers to check whether from a construction viewpoint the studied improvements were feasible and, in particular, to what extent. Manufacturers confirmed the weldability of the tubes to the tube-sheet and defined limits for the length of the tube protrusions.

The design was also updated by switching from internal to external ferrules, as it was no longer possible to maintain an internal ferrule without reducing the passage area inside the tubes. The ferrules were also subject to other modifications aimed at improving the sealing over the tubes and their stability; based on feedback from manufacturers and end-users, the design of the hold-down grid for the ferrules was also updated to improve its duty and to facilitate its installation.

Another aspect, which is a high priority for end-users worldwide, is the importance of inspections for proper maintenance of the equipment: a well scheduled inspection and maintenance plan for the equipment is of fundamental importance to meet the current practice of having turn-arounds every three or even four years.

Saipem recommends that the maintenance of the HP equipment should be performed by specialised and qualified companies (including, but not limited to, the equipment manufacturers) so that the quality of the repair activities is ensured with a beneficial effect on equipment life. By hiring specialised contractors with a deep knowledge of the equipment to attend the maintenance, the risk of catastrophic failure due to a repair activity not being performed properly is minimised.

It is important to note that equipment failure can have dramatic consequences, firstly from a safety viewpoint considering

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the operating conditions (temperature and pressure) and the toxicity of the fluids circulating in the equipment and, secondly, from an economic point of view considering the loss of production while the equipment is repaired (even if temporary) or replaced.

Maintenance is of particular importance for equipment close to the expected end of life where each small repair and maintenance activity can lead to important extensions of equipment life. The lack of appropriate inspection and maintenance is what has led to the failure of some older reactors in recent years that were close to the end of their expected life.

During the investigation into the causes leading to failures, Saipem was able to identify a series of common causes, different in nature, but all leading to the final failure of the reactors.

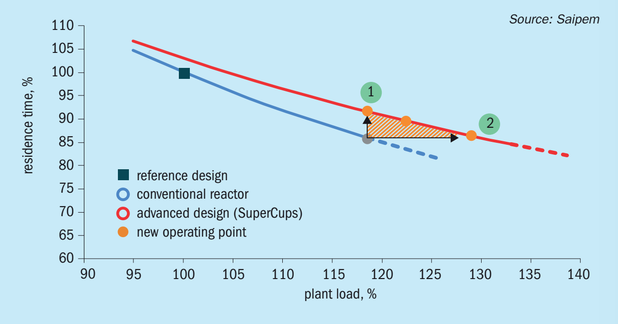
One of the main causes was the lack of appropriate inspections and data collection. Apart from the frequency and thoroughness of the inspections, it is important that proper and reliable measurements (e.g., for thickness) are taken using the correct tools, which should be properly handled and calibrated.

Another cause was the selection of a non-specialised contractor to attend the repair activity which, as already discussed, is a risk. It is of utmost importance that the contractor selected by the end-user, while performing the repair job, is fully aware of the design and characteristics of the equipment and of the consequences of a poorly executed repair job. It was also noted that proper supervision of the repair activities and the correct processing of the errors that occurred during the repair activity could have avoided the failures. For these reasons Saipem, as urea licensor, provides assistance and support services for the review of the procedures, qualifications, and drawings to be used during maintenance activities and invites all end-users to take advantage of such services with the aim of minimising the occurrence of issues during the execution of the activities they carry out themselves.

It is worth saying that, while it is the course of action by inspection and maintenance staff, being actively involved during the turnarounds, that probably has the most impact on prolonging the life of equipment, the operation team also has a share of the responsibility. The plant operators working in the plant every day are in fact best placed to be the first to detect possible leakages by performing a correct inspection of the weep holes. Weep holes are the mechanical expedients with the function to detect any leak from the lining welds (the welds performed between the different lining sectors) by providing passage of the leak across the pressure retaining wall of the equipment, via dedicated grooving and tubing, thus saving the equipment from carbon steel corrosion issues. The inspection of weep holes must be performed regularly, on a daily basis, taking care to also periodically verify that the weep hole circuits are free from plugs and, if any plug is found, to carefully remove them. Considering the total number of weep holes and their location on the plant, many end-users opt for an automatic leak detection system which ensures continuous monitoring, sparing the operators the time required to check the weep hole, allowing them to attend other activities providing better time management.

Various automatic leak detection systems are available on the market, proposed by different vendors and having different operating schemes and working principles (e.g., operating under vacuum or pressurised conditions). Saipem advocates the use of automatic leak detection systems provided that they are designed considering the peculiarities of the urea environment with particular attention to avoid the formation of plugs and to ensure that leaks are promptly detected. Saipem has not developed its own proprietary solution, preferring instead to rely on a proven and qualified solution already available on the market. This solution, based on the vacuum operating principle, has been jointly developed and commercialised by renowned and esteemed companies and it was technically approved by Saipem, after a thoughtful review involving both its capabilities as licensor of Snamprogetti™ urea technology and as EPC contractor.

Fig. 1: Improvements in reactor performance with SuperCups



To conclude, Saipem encourages end-users to exploit the scheduled maintenance activities and/or the replacement of equipment to boost the performances of their plant(s). Replacing equipment that has reached end of life with the latest updated design of equipment can also provide an opportunity to debottleneck the plant e.g. by increasing the exchanger surfaces or by increasing the reaction volumes. Variations to exchanger surfaces and volumes may, however, be limited by structural constraints. In fact, a bigger piece of equipment may not have sufficient space due to existing structures, piping and other equipment or it may require modifications to the structures or foundations due to the increased weight.

A solution to this for both existing reactors and new replacements is to install SuperCups. By means of their triple fluid-dynamic effect, SuperCups can easily boost the performance of an existing plant by promoting contact of the reagents and enhancing reactor conversion. It has been demonstrated that the characteristic "plant load vs residence time" curve of a reactor installed with SuperCups is higher than one with sieve trays (Fig. 1). By switching from sieve trays to SuperCups, the residence time of the reactor for a fixed capacity is increased allowing higher conversions rates to be achieved. Higher conversion rates in the reactor mean lower decomposition (primarily in the urea stripper), condensation (e.g., in the carbamate condenser) and recycling loads to the downstream equipment, enabling the plant to reduce its power consumption, and therefore its carbon footprint, or to increase its production without further and more expensive interventions. ■

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TOYO

Technologies for improved reliability and safety of urea plants

K. Yoshimoto

Toyo Engineering Corporation (TOYO), a global leading engineering contractor and urea process licensor, has developed various technologies to improve the reliability and safety of urea plants and increase efficiency. In this article, TOYO introduces its innovative technology line-up:

- super duplex stainless steel (DP28W™);
- leak detection system;
- AOCM™ (Advanced Online Corrosion Monitoring);
- IDCS™ (Image Diagnosis for Corrosion Screening).

Amongst others, AOCM™ and IDCS™ are recent introductions which TOYO believes will enhance maintenance activities for the critical equipment in urea plants.

DP28W™

TOYO has more than 40 years of experience in applying duplex stainless steels to urea plants. The most significant advantage of duplex stainless steel is its excellent corrosion resistance in urea-carbamate solution, which enhances the reliability and safety of equipment, and reduces the amount of passivation air required. Responding to industry demand, TOYO and Nippon Steel Corporation (NSC, formerly Sumitomo Metal Ind., Ltd) developed an advanced duplex stainless steel for urea plants, named DP28W™, which has greatly improved the safety and economy of the urea plant over its lifecycle.

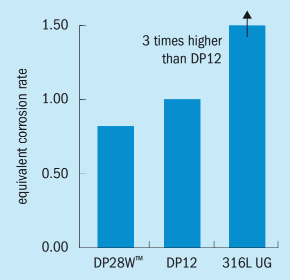
Features of DP28W™

DP28W™ has outstanding features in terms of alloying design, mechanical properties and corrosion resistance.

Alloying design

DP28W™ (28Cr-8Ni-1Mo-2W) shows excellent corrosion resistance, not only in the base metal, but also in the heat-affected zone (HAZ). A combination of the high chromium content, the addition of tungsten and a well-balanced ferrite/austenite structure have achieved excellent corrosion resistance. The molybdenum content is intentionally reduced to keep good corrosion resistance in the HAZ. The addition of tungsten compensates for the adverse effect on corrosion resistance in the base metal by reduced molybdenum. Optimisation of the chemical composition has been done by multiple linear regression analysis with the data obtained through tests in a pilot plant and commercial plants.

Fig. 1: Material corrosion rate comparison



Source: TOYO

Mechanical properties

DP28W™ has remarkable mechanical properties. Table 1 shows an example of tensile test results for DP28W™ compared to other stainless steels. The table indicates that DP28W™ has high mechanical strength compared to austenitic stainless steels such as 25Cr-22Ni-2Mo and 316L, providing a great advantage for the mechanical design of equipment.

Corrosion resistance

Corrosion resistance of DP28W™ to urea-carbamate solution has been proven in commercial plants. An example of immersion test results in a urea stripper is shown in Fig. 1. As shown, 316L UG corrodes at a high rate because it cannot maintain the passivation film due to severe corrosive conditions in the stripper. By contrast, DP28W™ shows excellent corrosion resistance, and also provides a lower corrosion rate than DP12.

Advantages of DP28W™ over the plant lifecycle

DP28W™ provides a number of great advantages which contribute to the safety and economy of the urea plant:

Enhanced reliability and maintainability of equipment

The expected corrosion rate of DP28W™ is lower than existing materials. The corrosion resistance in the HAZ and weld metal is greatly improved compared to conventional duplex stainless steels. These positive features provide the following advantages over the plant lifecycle:

- prolongs the life of components exposed to urea-carbamate solution;
- decreases maintenance frequency;
- reduces the risk of active corrosion.

Decrease of passivation air

DP28W™ is easily passivated with less dissolved oxygen in urea-carbamate solution. It suggests that the passivation air, which restricts operational efficiency due to lower conversion in the synthesis section and ammonia loss in the recycle section, etc. can be positively reduced.

Design allowance

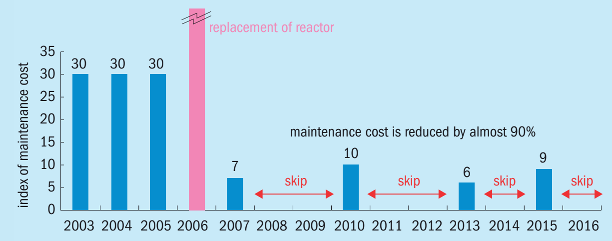
DP28W™ has a higher allowable stress value and stress intensity value than austenitic stainless steels and conventional duplex stainless steels. This high mechanical strength provides a larger design allowance.

Table 1: Tensile test results for DP28W™ and other materials

	Tensile strength, MPa	Yield strength, MPa	Elongation, %
DP28W™	934	647	42
DP12	822	610	42
25Cr-22Ni-2Mo	676	352	50
316L	518	234	52

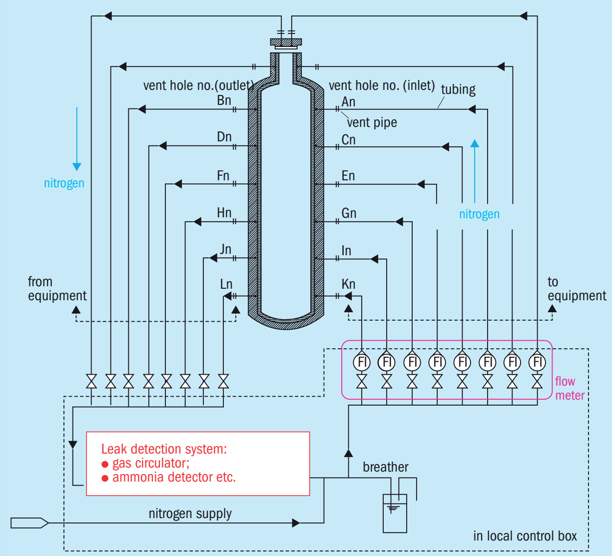
Source: TOYO

Fig. 2: Index of maintenance cost before and after reactor replacement



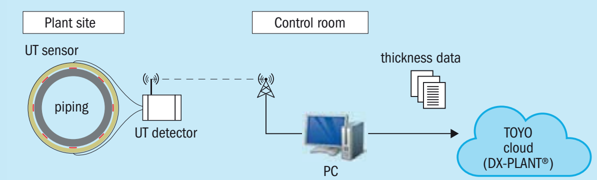
Source: TOYO

Fig. 3: TOYO's leak detection system



Source: TOYO

Fig. 4: AOCM™ system



Source: TOYO

Experience

DP28W™ has been applied to TOYO urea plants since the middle of the 2000s and has shown excellent performance to date. For example, one owner replaced its existing titanium-lined urea reactor, in the total recycle process, with a new reactor lined with DP28W™. The owner had clear intentions to operate the new reactor under the same operating conditions as the existing reactor (200 bar, 200°C). The operating condition was severe as titanium requires little passivation air even at high temperature. Nevertheless, it is notable that the actual corrosion rate of DP28W™ has been far lower than titanium. The new reactor fabricated with DP28W™ has been operating successfully since it was put into operation in 2006 and the owner has reduced its maintenance cost drastically by 90% as shown in Fig. 2. The maintenance activity during each turnaround has been mainly visual inspection only and there have been no major repairs caused by the material.

Leak detection system

The internal wall of the high-pressure equipment in the urea plant is in contact with corrosive fluid and is lined by a proper corrosion-resistant metal plate. In the event that this lining plate has a leakage, it leads to damage to the pressure holding shell made of carbon steel.

TOYO has developed an on-line leak detection system by detecting ammonia leaking in circulating nitrogen gas between the lining plate and pressure holding shell (See Fig. 3). This system has the advantage of being able to detect tiny amounts of leakage and to identify the location of the defect before the damage becomes severe.

The system also contributes to making maintenance work easy and simple, and increases the reliability of the urea plant.

AOCM™ (advanced online corrosion monitoring)

Corrosion monitoring of high-pressure equipment and piping in the urea synthesis loop on a real-time basis is beneficial for plant owners not only in establishing optimised inspection plans, but also enabling reliable operation. Assessing a real-time condition of stainless steels exposed to ammonium carbamate is particularly challenging. AOCM™ is a solution that can predict the condition of the equipment and piping.

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Fig. 4 shows the AOCM™ system applied to synthesis piping. Ultrasonic testing (UT) sensors installed in the piping measure the corrosion rate on a real-time basis.

AOCM™ application to ACES21®

TOYO has developed AOCM™ to apply to ACES21®, TOYO's urea synthesis technology (see Fig. 5).

Piping

AOCM™ is applied to the piping in the ACES21® synthesis section, where its main function is to monitor the corrosion rate of the piping on a real-time basis. UT sensors are installed directly on the piping in various locations. The piping thickness measured by UT sensors is transferred to DX-PLANT® wirelessly on a real-time basis; this allows plant owners to monitor corrosion rates of individual piping with reference to operational parameters such as temperatures in real time.

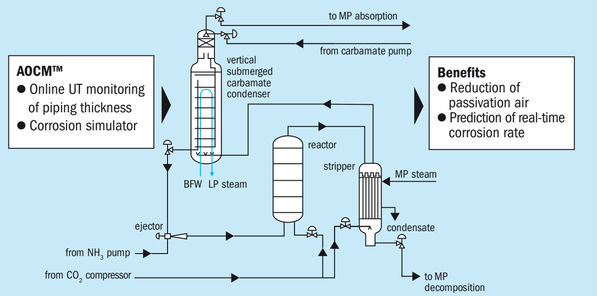
Equipment

The corrosion simulator has been developed by TOYO based on empirical data obtained from immersion tests and is utilised for on-line corrosion monitoring of high-pressure equipment, since the installation of UT sensors in such high-pressure equipment is physically impractical. TOYO's corrosion simulator predicts the corrosion rates of each type of stainless steel for both the equipment and the piping in the urea synthesis section using operating temperature and oxygen concentration in CO_2. As there is a certain correlation between the corrosion rates of the equipment and the piping, AOCM™ compares the corrosion rates obtained by the corrosion simulator and the UT sensors to provide a more accurate corrosion rate of the equipment.

Benefits of AOCM™ for plant owners

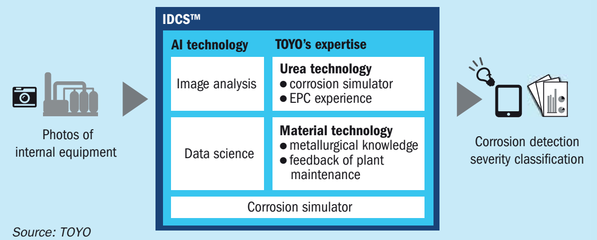
Since the urea synthesis section is a highly corrosive environment, passivation air needs to be introduced for corrosion prevention. Optimisation of the amount of passivation air will bring considerable benefits for plant owners, for example, increasing the urea synthesis rate or saving energy consumption, leading to opex reduction. While insufficient passivation air will cause severe metal loss due to active corrosion, AOCM™ ensures that passivation air can be reduced by corrosion monitoring on a real-time basis without the risk of active corrosion. Furthermore, plant owners can optimise shutdown maintenance activities and shutdown schedule (interval, period, timing) in advance by using the accurate information on corrosion rates.

Fig. 5: **Application to ACES21®**



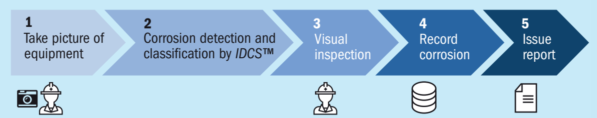
Source: TOYO

Fig. 6: **Outline of IDCS™**



Source: TOYO

Fig. 7: **Workflow of new methodology of inspection with IDCS™**



Source: TOYO

IDCS™ (image diagnosis for corrosion screening)

TOYO's image diagnosis for corrosion screening (IDCS™) system is a new way to screen and detect corrosion of equipment internals during plant maintenance. This system utilises image analysis technology by artificial intelligence (AI) and enables efficient inspection for equipment during plant maintenance. Fig. 6 shows an outline of IDCS™. The system is configured with a combination of AI technologies, such as image analysis, data science, a corrosion simulator and TOYO's expertise, enabling a high speed and reliable analysis. IDCS™ facilitates corrosion detection classification of the severity for equipment internals using image data of the target equipment, as described in the following section.

The advantages of IDCS™ are:

- Time saving, accurate, and less person-dependent visual inspection during plant maintenance:
  - IDCS™ provides greater efficiency compared to conventional inspection in which inspectors spend a long time on a 'one-by-one' visual inspection and the quality of the inspection tends to be highly dependent on the inspectors' skills and time available.
- Dependable and easier estimation of timing for repair or replacement of equipment
  - By applying IDCS™ periodically on the plant inspection and accumulating the historical data obtained, condition-based maintenance with reduced opex can be realised.

Work flow of new methodology of plant maintenance with IDCS™

Fig. 7 shows a work flow of the new methodology of inspection utilising IDCS™ during plant maintenance. The procedure includes the following steps:

- take pictures of equipment internals and import them to IDCS™;
- IDCS™ detects corrosion and classifies corrosion severity into 4 levels by image analysis of the pictures;
- inspection personnel perform visual inspection mainly where the system detected corrosion;
- automatically record the corrosion details (location and severity of corrosion);
- automatically issue reports including the corrosion details.

IDCS™ verification test

At the development phase, TOYO conducted a verification test of IDCS™ for the stripper in the urea synthesis section.

The stripper has been operating for over 20 years in some TOYO ACES plants (previous process to the latest ACES21®) and corrosion by ammonium carbamate has been occasionally observed on the tube-to-tubesheet (T/TS) welds. Corrosion on the weld HAZ on tube internals was the most inspected point. In addition, conventional visual inspection of thousands of T/TS welds requires highly skilled personnel and takes a long time. Therefore, utilising the sample of the corrosion on tube internals, the verification test was conducted to validate if IDCS™ has the potential to be applied to the actual equipment for time-saving and less person-dependent inspection.

Table 2 shows models of defined corrosion of T/TS weld internals for different levels of severity. At level 0 there is no abnormality. At level 1, colour change is confirmed at the welded part. At level 2 slight thinning is seen on the weld heat affected zone and periodic observation is required for levels 1 and 2. Severe thinning is confirmed on a wide area at level 3. This must be repaired as soon as it is detected.

In the first step of the IDCS™ verification test, the IDCS™ had to manually learn hundreds of pictures of reference T/TS welds for each level as training data sets. After this stage had been completed, IDCS™ automatically classified the condition of other T/TS welds according to the four levels of severity and marked the corroded tubes, indicating their level. Fig. 8 shows an example of the output image obtained by IDCS™. The accuracy of the classification of the corroded T/TS weld internals was higher than 80%. Further improvement to the accuracy is ongoing, aimed at application for actual equipment.

Table 2: **Models of defined corrosion severities**

Severity	Level 0	Level 1	Level 2	Level 3
Appearance				
Remarks	No abnormality	Colour change at welded part	Slight thinning (marked area)	Severe thinning (marked area)
Required action	N/A	Periodic observation	Periodic observation	Repair weld

Source: TOYO

Fig. 8: **Output image of IDCS™**

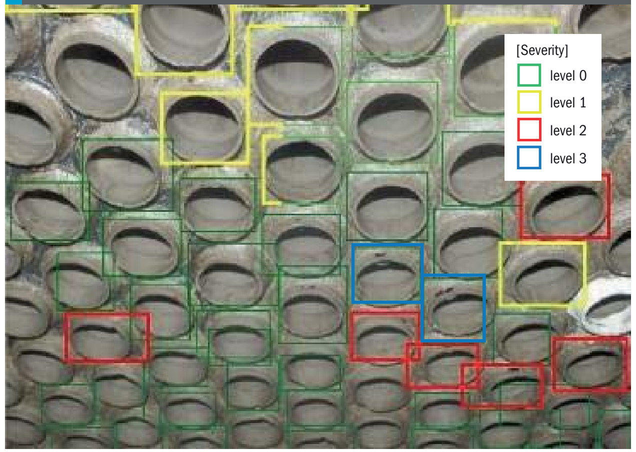


PHOTO: TOYO

Further development of IDCS™

Considering the results obtained from the verification test, IDCS™ is expected to be applicable to various kinds of plant equipment, regardless of the type of process. To expand the areas of application, some further development is being carried out in the following areas:

- application to weld lines (ex. lining welds, support ring welds);
- application to corrosion cracking, localised corrosion, and weld defects;
- sizing of corrosion and damage;
- identification of the inspected parts (ex. rows and numbers of hex. tubes);
- application to movies recorded in equipment during inspection;
- application to wearable cameras for real time inspection. ■

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STAMICARBON

Boosting reactor performance by high-efficiency trays

M. Beaujean and A. Shams

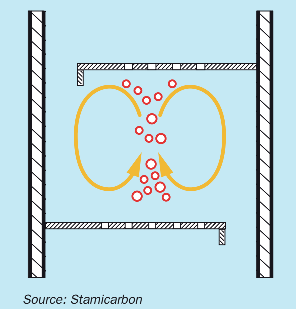
In all commercial processes, urea is produced by reacting ammonia and carbon dioxide at elevated temperature and pressure in the urea reactor and can proceed up to chemical equilibrium. Because of the equilibrium nature of the urea reaction, the reaction is preferably done in a plug flow reactor.

This conversion in the urea reactor is mainly determined by the residence time, reactor volume and sufficient contact between the gas phase and the liquid phase.

Generally speaking, a plug flow reactor can be achieved by installing numerous continuously stirred tank reactors (CSTR) in series, which can substantially improve the reaction kinetics. Practically this can be realised by installing a certain number of sieve trays along the reactor length, which divide the reactor into several compartments. In this way, the reactor turns into a bubble column with a torus circulation, which improves the contact between the gas and liquid and increases the urea conversion accordingly. The liquid risers are located at a 180° staggered position, to ensure a zigzag flow pattern. This flow pattern will increase the path of the fluids along the reactor and hence increases the residence time and urea conversion (Fig. 1).

The condensation of ammonia and carbon dioxide causes the quantity of gas to decrease and the temperature to increase, during passage through the reactor. A gas cushion below each tray with a certain height is considered in the design to ensure sufficient seal between the gas and liquid and to avoid the backmixing effect, which reduces the urea conversion in the reactor. The sieve trays in the top of the reactor have fewer holes than those in the bottom. This ensures that also in the top of the reactor, the cushion of gas present under the sieve trays will avoid backmixing and thus maintain the plug flow regime. A plug flow in the reactor is a theoretical assumption as plug flow means there is no backmixing at all. In practice some degree of backmixing is inevitable.

Fig. 1: **Torus circulation in reactor compartments**



Source: Stamicarbon

Reactor issues

Stamicarbon, the innovation and licensing company of Maire Tecnimont Group, has developed high-efficiency reactor trays, which have been installed in many urea plants. These trays improve the conversion in the reactor substantially and can solve the common issues which most urea reactors suffer from. These common issues can be summarised as follows:

Backmixing

Backmixing (Fig. 2a) occurs when the liquid accidentally passes through the gas holes. That normally occurs when no or insufficient gas cushion underneath the tray is present. The high efficiency reactor trays as designed by Stamicarbon have sufficient gas cushion to avoid backmixing from occurring. Backmixing can also occur as a result of improper levelling of the tray during the installation which may break or disturb the gas cushion underneath the tray.

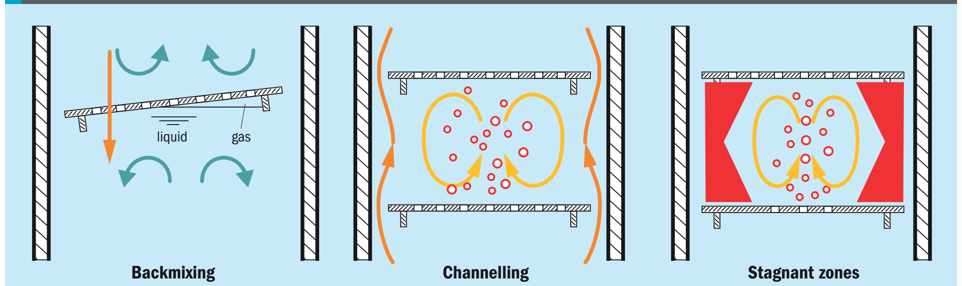
Channelling

Channelling (Fig. 2b) normally occurs in reactors with no internals or with conventional trays. Channelling occurs when liquid by-passes compartment without having proper contact with the gas. As a result, low conversion in the reactor is expected. This issue can be excluded in the high efficiency reactor trays with 180° staggered flow path in which a zigzag flow pattern with an optimum gas-liquid contact is ensured.

Stagnant zones

Creating stagnant zones in the reactor (Fig. 2c) can be an issue for the conversion due to the poor contact between the liquid and the gas, which affects the urea conversion consequently. This issue is also solved with the high-efficiency reactor trays by having the zigzag flow pattern, which avoid stagnant zone formation.

Fig. 2: **Common issues in urea reactors**



Source: Stamicarbon

Fig. 3: Tray in SPIC reactor.

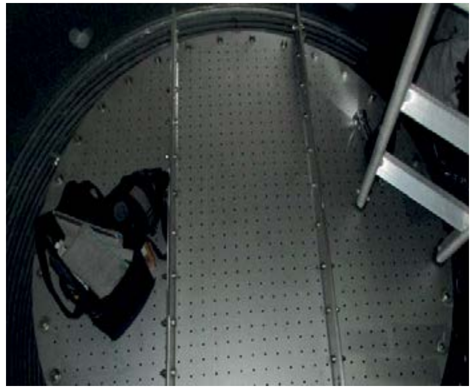


Fig. 4: Mixing tray.

PHOTOS: STAMICARBON

General reactor performance

The high-efficiency reactor trays will increase the conversion in the reactor and hence lower the load on the high pressure stripper and high pressure carbamate condenser. This means that the process limits in these equipment items are encountered at a higher plant capacity. The improved reactor conversion is demonstrated by a reduced steam consumption of the high pressure stripper along with a reduced steam production from the high pressure carbamate condenser. Apart from saving energy on the high pressure stripper, the high-efficiency reactor trays may also be used to increase the capacity of the urea plant. The main parameters in the synthesis section are the steam consumption to the high pressure stripper, steam production from the high pressure carbamate condenser, N/C ratio in the reactor overflow, CO_2 conversion and top temperature of the reactor.

High-efficiency reactor trays experience

In 2017, Southern Petrochemical Industries Corporation Ltd (SPIC) in India requested that Stamicarbon replace their existing HP reactor with a new one in Safurex®. The old reactor was already at the end of its life time with some major repairs necessary, due to excessive leakages occurred during its operation time. The urea plant was licensed in 1974 and uses TOYO technology with a nameplate capacity of 1,600 t/d. With the old reactor the plant could achieve a capacity up to 2,080 t/d. There were a lot of process inefficiencies due to aging of the equipment, which have had a negative impact on the production and overall performance of the plant.

Reactor features

The old HP reactor was a plug flow reactor with no internals. The outer shell of the reactor was made of carbon steel, while the liner in contact with the process was made of titanium. For the reactor replacement Stamicarbon advised an improved conversion design, including high-efficiency trays and other internals made of Safurex®.

After delivery of the new Safurex® reactor to client's plant site in 2019, Stamicarbon performed supervision services during installation of the reactor including final inspection. After start-up, Stamicarbon gave process support and training to the operational staff, regarding the features and improved performance figures of the new reactor.

Since the diameter and length and thus the reactor volume of the new reactor were kept the same, it offered Stamicarbon the opportunity to compare and evaluate the performance of the reactor before and after the replacement. Any improvement in the conversion can be attributed to the introduction of the high-efficiency trays with hardly any modifications in the downstream section. A complicating factor was the fact that in this plant there was no sample point downstream of the reactor.

In the SPIC plant, a conventional process is applied in which all the reactants (ammonia, CO_2 and carbamate) are added to the reactor. The reactants are present in different phases before being introduced to the reactor. Ammonia and carbamate are in the liquid phase while the CO_2 is in the gas/supercritical phase. To achieve proper mixing of these reactants, an inlet gas/liquid mixer has been designed.

Process evaluation before replacement

In order to visualise the differences before and after the reactor replacement, a Stamicarbon model was built to replicate the existing situation before the reactor replacement. The current balance was used as a starting point to prepare a new balance after installing the high-efficiency reactor trays. According to the mass balance, the following improvements were expected:

- CO_2 conversion would increase from 59.4% to about 61.2%;
- the urea content in the reactor outlet stream would increase from 32.5% to about 33.5%;
- the amount of carbamate recycle would reduce from 103.3 m³/h to 97.0 m³/h;
- due to the higher urea conversion in the reactor, less carbamate would end up in the downstream section. The heat required to decompose the carbamate to gaseous CO_2 and NH_3 would decrease. The steam consumption was expected to be reduced to approximately 4% compared to the current steam consumption.

The predicted promising model figures resulted in the following expectations:

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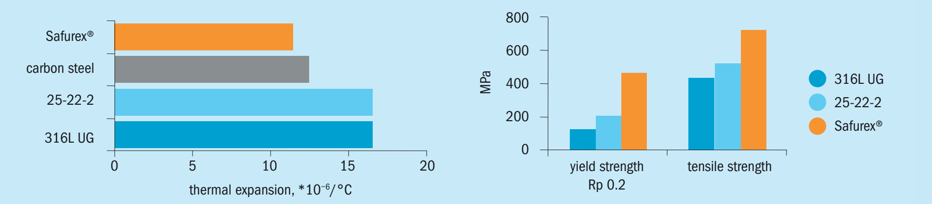
Fig. 5: **Thermal expansion coefficient, yield and tensile strength of Safurex® compared to other steel material**



Source: Stamicarbon

- an increase in the operational flexibility and a reduction in the maintenance cost;
- less steam consumption;
- higher plant capacity preconditioned by feedstock availability and unlimited utility.

Situation after replacement

After the reactor replacement and plant start-up some operational parameters were collected during a plant visit, to verify the figures claimed by the model. The main drive was to ensure that the new high-efficiency reactor trays delivered the promised added value (Figs 3 and 4).

The plant load achieved a capacity of 5% above the previously reached capacity with the old reactor and increased from 2,080 to 2,180 t/d. One of the main influencers that helped to increase the plant load was the significant reduction in the carbamate recycle. According to the original situation around 103 t/h was recycled back to the reactor. According to the SPIC operation team, this carbamate recycle rate had fallen in recent years to 90 t/h. After the reactor replacement, due to the higher urea conversion, a carbamate recycle as low as 78 t/h could be achieved, a reduction of 13%.

Based on the lower carbamate recycle, a lower steam reduction was to be expected. After starting the plant a steam reduction of about 4% could be achieved, as predicted by the Stamicarbon model. The reduction in steam consumption is considered a great benefit in terms of opex. The steam consumption can be improved further by increasing the efficiency of the downstream section.

Since the material of construction of the liner (wetted parts) was changed from titanium to Safurex®, operation at a lower passivation air flow rate is allowed. This flow reduced about 38% compared to the old reactor. This allows the reduction of inerts in the reactor, increases the reactor outlet temperature and consequently increases the urea conversion in the reactor.

Safurex® as material of construction

The main advantage of using Safurex® in the extremely high corrosion urea-carbamate environment can be summarised in the following:

- due to the ferrite austenite microstructure, the sensitivity for stress corrosion cracking by chlorides can be considered as limited;
- no or minimum amount of passivation air is required to prevent the active corrosion;
- longer inspection interval, thus less inspections and more production output due to the fact that there is no risk of active corrosion and thus increasing the mechanical integrity of the equipment;
- superior properties in term of corrosion resistance and high mechanical strength in combination with low thermal expansion. The thermal expansion coefficient of Safurex® is almost identical to that of carbon steel as indicated in Fig. 5. Equipment in Safurex® therefore has a much lower risk on buckling or other types of deformation due to differences in thermal coefficients;
- it is not prone to strain induced intergranular cracking. Thus, the heating rate of the reactor is less strict compared to the old reactor. The heating rate of the reactor increased from 50°C to 60°C in the new reactor;
- it is not prone to active corrosion. Block-in time consequently can be extended, as long as the reactor wall temperatures are above 125°C, before the necessity of having a complete drain or re-passivation. Of course the mechanical integrity and the applicability of the non-Safurex® piping attached to the reactor in the HP section should be checked for the long block-in time;
- high reliable construction material with a proven track record for more than 25 years used in more than 200 items of all kinds of high pressure equipment.

Conclusions

With this operational example, Stamicarbon could quantify the added value of installing the high-efficiency reactor trays in non-Stamicarbon plants. The benefits of installing these trays in this specific plant could be clearly observed due to changing from no trays to high-efficiency reactor trays.

The process benefits of installing the high-efficiency reactor trays in this project can be summarised in:

- higher urea conversion
- less carbamate recycle
- lower steam consumption
- higher plant output

The mechanical benefits of using Safurex® as a material of construction for the liner are:

- to increase the mechanical integrity of the equipment by selecting steel with high strength;
- to eliminate many of the corrosion behaviour/failure modes, such as stress corrosion cracking and stress induced intergranular cracks;
- to lower the O_2 content in the plant;
- the ability to block-in the plant for longer periods as long as the temperature is above 125°C and the other interconnecting pipes allow, plus more flexibility in term of start-up and heating up the equipment. ■

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Safe handling and start-up of ammonia synthesis catalyst

Ammonia synthesis catalysts have long lives and catalyst replacement is an infrequent activity. Many people will go through their careers in the ammonia industry without ever having to replace a synthesis catalyst and the infrequent nature of catalyst replacement means that many plants may not have direct experience of this activity. Ammonia synthesis catalyst can present a range of hazards throughout the replacement process, from transport through loading, reduction, start-up, shutdown and discharge, but the good practice illustrated in this article, and collaboration between catalyst suppliers and end users can ensure safe and successful catalyst changeouts.

JOHNSON MATTHEY

Good handling practice for safe and successful catalyst changeouts

J. Brightling, J. Pach and T. Ithell

With the exception of a few plants which use a ruthenium based catalyst, all ammonia synthesis catalysts in commercial operation use promoted iron oxide, which is then reduced to promoted iron prior to use – broadly similar to that developed by Mittasch in the early part of the 20th Century. Whilst it may seem that there has been little subsequent development, this is deceptive in that there is now a much greater understanding about the role of the various promoters and how to optimise their incorporation during catalyst manufacture.

Promoters can be categorised as structural or electronic. Some are added deliberately during production whilst others are found in the iron ore used to produce the catalyst. Electronic promoters increase the activity of the catalyst by changing the electronic nature of the catalyst surface. K_2O is the most important electronic promoter. Structural promoters act to maintain the active structure of the catalyst, in particular its surface area, under normal operating conditions. Alumina and calcium oxide are the most important structural promoters.

There is considerable interaction between the various promoters, and this can have a dramatic effect on initial reducibility, initial activity, and long-term stability. A thorough understanding of these interactions is vital in order to optimise the performance of the catalyst and to understand the safe handling of the material.

KATALCO™ series catalysts

A major improvement to iron-based ammonia synthesis catalysis was the development of KATALCO™ 74-1 in 1984. Although over 35 years old, this catalyst remains the highest activity iron-based catalyst available in the market. It was initially developed for use at low pressure in ICI's AMV and LCA plants. More recently its high activity has been integral to the success of the Uhde dual pressure process. The main difference between KATALCO 74-1 and standard iron-based catalyst is the incorporation of cobalt. A unique manufacturing process ensures optimum performance. In addition to high activity, ease of reduction is a significant benefit.

Although KATALCO 74-1 is supplied in oxide form, metallic iron is the active form for ammonia synthesis and the iron oxide needs to be reduced prior to use by reacting with hydrogen:

$$Fe_3O_4 + 4H_2 = 3Fe + 4H_2O$$
$$\Delta H_R(25°C) = +609 \text{ kJ/kg}$$

As the reaction is endothermic at low temperature, a source of heat is required to initiate the reduction.

A well-executed reduction takes place with a sharp and well-defined reaction front passing through the converter. Upstream of the reaction front the catalyst is reduced, and hence active. Downstream of the reaction front is unreduced catalyst with little ammonia synthesis activity. In most cases the source of the hydrogen is methanated synthesis gas. The presence of nitrogen in near stoichiometric quantities is beneficial because ammonia synthesis can occur over the active catalyst.

Water acts as an irreversible poison for ammonia synthesis catalyst during reduction and water that is generated must be kept to a low level. This is achieved by limiting the concentration of water to a level specified by the catalyst vendor (typically 1,000 to 3,000 ppm) and by performing the reduction at a low pressure to minimise the partial pressure of water.

Plant reduction can be a lengthy and costly process and in many cases it is the operational problems due to the plant (start-up heater / circulator / converter designs) rather than the catalyst that is the rate determining step.

If the catalyst is indeed the limiting step, an alternative is to use a catalyst with good reduction properties such as KATALCO 74-1.

Another good alternative is the use of KATALCO 35-8 or KATALCO 74-1R, which have been reduced and then passivated by carefully regulated exposure to oxygen to give a stable iron oxide skin around the active iron phase. Reduction of this skin is much easier (and faster) than reduction of bulk catalyst allowing a substantial decrease in the time required for activation.

The disadvantages with pre-reduced catalyst are a higher cost and the extra care that is required when handling the material. A common compromise is to use a mixture, with a small quantity of pre-reduced catalyst on top of a larger volume of standard catalyst.

KATALCO 35-8A
SAFETY DATA SHEET
Conforms to Hazard Communication Standard 2012 (HCS 2012)

Section 14. Transport information

	DOT Classification	TDG Classification	Mexico Classification	ADR/RID	IMDG	IATA
UN number	UN2881	UN2881	UN2881	UN2881	UN2881	UN2881
UN proper shipping name	Metal catalyst, dry (iron)	Metal catalyst, dry (iron)	Metal catalyst, dry (iron)	Metal catalyst, dry (iron)	Metal catalyst, dry (iron)	Metal catalyst, dry (iron)
Transport hazard class (es)	4.2	4.2	4.2	4.2	4.2	4.2
Packing group	II	II	II	II	II	II
Environmental hazards	No.	No.	No.	No.	No.	No.

SOURCE: JOHNSON MATTHEY

Fig. 1: KATALCO 35-8A ammonia synthesis catalyst transport classifications

KATALCO 35-8A
SAFETY DATA SHEET
Conforms to Hazard Communication Standard 2012 (HCS 2012)

Section 2. Hazards identification

OSHA/HCS status : This material is considered hazardous by the OSHA Hazard Communication Standard (29 CFR 1910.1200).
Classification of the substance or mixture : SELF-HEATING SUBSTANCES AND MIXTURES - Category 1
SERIOUS EYE DAMAGE - Category 1

GHS label elements
Hazard pictograms :
Signal word : Danger
Hazard statements : Self-heating; may catch fire.
Causes serious eye damage.
Precautionary statements
Prevention : Wear protective gloves and eye/face protection. Wear eye or face protection. Keep cool. Protect from sunlight.
Response : IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER or physician.

SOURCE: JOHNSON MATTHEY

Fig. 2: Extract of Safety Data Sheet for KATALCO 35-8A pre-reduced ammonia synthesis catalyst.

Catalyst handling

Catalyst transport

Catalysts are by design very active materials, for some products especially where they are pre-treated this brings to bear additional transport restrictions. Fig. 1 provides an example of the transport classification for pre-reduced KATALCO 35-8A ammonia synthesis catalyst on a safety data sheet.

Normally, for planned catalyst deliveries, sea freight is used. There are restrictions on what can be safely and legally transported using air freight, as a pre-reduced catalyst falls under Transport hazard class 4.2 "Spontaneously combustible substance", since the catalyst can self-heat (without exposure to a heat source) when exposed to oxygen. Practically, this means it is not usually possible to ship pre-reduced ammonia synthesis catalyst by air.

This relevant transportation information is provided on the MSDS/SDS, an extract from which can be seen in Fig. 2. Additionally, there is a requirement to label each package with a HAZ label.

Catalyst storage

KATALCO series ammonia synthesis catalyst is normally supplied in mild steel drums. These drums must not be stacked on their sides or stacked more than four drums high, even when held on pallets. Stacking of drums introduces both the risk of top drums falling from the stack, and the lower drums being crushed under the weight of the stack.

If the metal drums are to be stored outside, this should only be for a maximum of a few months, and they should be protected against rain and standing water. Longer term storage should be under cover and away from damp. To prevent contamination the lids should be left on the drums until just before charging.

Catalyst drums must not be rolled. Careless handling of the drums risks damage to the catalyst. Catalyst drums are often supplied on pallets, which reduces the likelihood of damage in transit, but requires suitable fork-lift trucks (Fig. 3) and a paved area to handle the pallets. The fork-lift truck to be used for unloading the pallets should be fitted with rim or body clamps to avoid damage to the drums. It is important not to use standard forks to lift the drums under the rolling hoops, as damage to the drums and catalyst is almost inevitable.

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PHOTOS: JOHNSON MATTHEY

Fig. 3: Forklift loading of drums of KATALCO™ series catalyst onto a road truck.

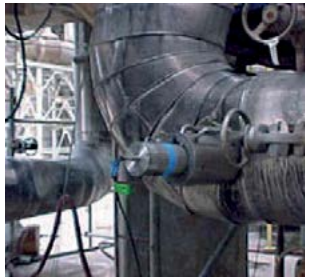

Fig. 4: Testing for ammonia converter internal leaks.

Pre-overhaul checks

Since replacement of the ammonia synthesis catalyst is an expensive and time-consuming activity with long periods between scheduled replacements, and maintenance in and around the converter is difficult once the catalyst is in its reduced state, it is worth taking care to ensure that the ammonia converter is returned to service in good condition after a catalyst change. If the cartridge is not being replaced during an overhaul, it is worthwhile analysing the performance of the converter to diagnose any potential issues which can be addressed during the overhaul.

Johnson Matthey can assist in performing such an evaluation using KATALCO Performance software and CFD modelling capabilities. If this proves inconclusive a Tracerco™ diagnostic check could also be carried out. This might involve injecting a radioisotope to check whether there is internal leakage through an ammonia converter (Fig. 4). These activities should be carried out sufficiently in advance of the overhaul to implement contingency plans should any defects be highlighted.

Catalyst installation

Once in the overhaul, and in addition to recommended mechanical inspections, typical pre-charging checks include:

- catalyst support grid integrity;
- cleanliness of internal heat exchangers;
- checking internal heat exchangers for leakage and possible bypassing (Tracerco process diagnostic techniques can again assist with this);
- quench and bypass valve operation;
- thermocouple positioning and operation (warm each in turn, check corresponding indication in the control room);
- ensure the vessel is dry before any catalyst is charged;
- clean any residual dust and debris from the catalyst beds prior to charging;
- ensure the catalyst is in good condition.

An even gas flow through the converter is key to ensuring optimum converter performance, therefore the catalyst loading method needs to ensure uniform bulk density and hence uniform pressure drop characteristics. This is especially important with radial flow beds to prevent voids in the catalyst bed and the resulting bypassing of gas. The density of ammonia synthesis catalyst is high and any equipment set up to handle the catalyst must be capable of withstanding its weight.

A rigorous and effective risk assessment should be carried out prior to catalyst charging. This should use advice from the catalyst supplier, the cartridge supplier and the plant designer. Protective clothing should be specified with due regard to the Material Safety Data Sheet (MSDS), local legislative requirements and operating company standards. As ever, special attention should be paid to the safety of confined space entries and to the asphyxiation hazards associated with nitrogen.

There is potential for the exposure to dust during catalyst loading. Catalysts should be handled in well ventilated areas and in manners that limit the formation of dust. Appropriate protective clothing, gloves and goggles should be worn, and respiratory protection should be used. All personnel involved in catalyst handling should wash afterwards, and clothing should be changed at the end of each shift as a minimum to prevent contamination.

Pre-reduced catalyst

Whilst there are many references of KATALCO 35-8 and KATALCO 74-1R being safely loaded into converters, there are a few features which should be considered when generating risk assessments and job methods. These are highlighted below.

If the oxide film around the reduced material is damaged, reduced catalyst is exposed to air. The reaction with air is highly exothermic and hence the catalyst particle heats up. As the temperature increases, the oxide film becomes a much easier barrier for oxygen to penetrate and hence a self-heating phenomenon occurs. Whilst a single particle has a large surface area and can transmit the heat of reaction away easily, in an ammonia converter, the heat of reaction is conducted to adjacent particles which then heat up, allowing oxygen to penetrate to the core of these particles and further increasing the temperature.

If there is no air flow through the packed bed, the concentration of oxygen falls as oxidation occurs and the reaction can no longer be self-sustaining and ceases. If oxygen is readily available, a temperature runaway is possible. As the density of the hot air in the bed decreases it will rise out of the bed, drawing in fresh air which allows the oxidation reaction to continue.

The protective oxide layer can be damaged by attrition and by moisture. The use of good quality catalyst, which does not require sieving, is recommended. Excessive vibration should also be discouraged. Excessive moisture can affect the protective layer and dehumidified air has also been used with success.

Additional precautions include keeping the catalyst cool (<50°C, 120°F) during handling. If exposed to hot tropical sun the catalyst can act as a black body and absorb the sun's heat until it becomes self-heating. Under such conditions, catalyst charging should be carried out under shade.

During catalyst charging, the vessel thermocouples must be fitted and operational to allow monitoring the temperature of the catalyst. If caught in time, the effects of catalyst heating can be accommodated by applying a nitrogen blanket. It is therefore necessary to have a readily available source of nitrogen and to keep a careful watch on the temperatures. The temperature should be monitored throughout the charging process until the converter is boxed up and under nitrogen.

If the catalyst reacts with air it will consume oxygen, and there is potential to generate an oxygen-deficient atmosphere during catalyst charging. It is recommended that the vessel is charged from outside if possible. If it is necessary to enter the vessel, breathing apparatus (BA) is recommended. Personnel entering the vessel must be fully aware of the possible hazards and a rescue plan should be established to enable them to leave the converter quickly and safely should temperature rise occur. Once it is safe to do so, a nitrogen blanket can then be established.

Standby personnel should also be wearing BA. BA should be used even if no nitrogen purge is activated since the pre-reduced catalyst will slowly consume oxygen within the confined space and may evolve some residual ammonia. No ventilation should be provided to avoid excess oxygen ingress promoting the exothermic oxidation reaction.

Reduction

Whilst the reduction procedure is quite simple, the effect of the synthesis loop means that a simple change in conditions takes much longer to settle down than would be the case with a once-through process.

Water evolved during reduction should never pass over catalyst that has been reduced, and this is achieved by keeping the unreduced beds at a lower temperature. The water concentration leaving the reactor should be controlled in accordance with the recommendations of the catalyst supplier.

The water is removed via the loop catchpot. This may be difficult at low loop pressure and it can freeze in the chillers if the refrigeration system is running. As ammonia solution freezes at a much lower temperature than water, ammonia is sometimes added to the loop before reduction commences. This means that the refrigeration system can be brought online earlier thereby minimising the water concentration in the circulating gas.

Gradually, the reduced catalyst at the inlet of the reactor will start to make more ammonia. At this stage, catalyst temperatures need to be controlled carefully (often by increasing the circulation rate), otherwise the reduction will become too rapid and may result in the catalyst temperature falling. Rapid temperature changes are always bad for a catalyst bed but, due to differential expansion, the catalyst is particularly vulnerable if it is partially reduced during such a change.

As the reduced catalyst produces ammonia, the reduction water contains an increasing level of ammonia. The ammonia liquor condensed from the reduction loop requires safe handling, storage and disposal.

As more catalyst is reduced, the heat of the ammonia synthesis reaction gradually becomes the dominant heat input. This allows the circulation rate to be increased tremendously, and so each bed takes roughly the same length of time to reduce even though the downstream beds contain much more catalyst.

The circulation rate is normally increased by opening the converter inlet valve a little. This directs more cool gas to the first bed and will cool/hold the bed temperature. It can also affect the flow through shot/bypass valves and therefore the flow through the various beds and interchangers. Adjustments to the inlet valve should be very small until the "feel" of the converter has been established. If a plant trip occurs a forward flow through the converter must be maintained by purging downstream of the converter to prevent reduced catalyst standing in a stagnant atmosphere containing water as this will reduce the final activity of the catalyst. If it is necessary to de-pressure the loop the catalyst should be held under a dry nitrogen atmosphere.

Shutdown

When an ammonia converter is taken offline it is important to exclude air, water and other possible catalyst poisons. The simplest way of doing this is to leave the loop at pressure, the only complication being that liquid ammonia may condense in sections of the pipework which are normally above the condensation temperature. If the pressure is subsequently reduced and the ammonia boils it may produce pipework temperatures below their design limits. A blow through with warm nitrogen or synthesis gas can avoid this problem.

In the right circumstances it is possible to reach temperatures much lower than the boiling point of ammonia (-33°C, -27°F, at atmospheric pressure) and, in the limit, it is possible to reach the adiabatic saturation temperature (-72°C, -98°F).

If the pressure in the loop is to be reduced to atmospheric, a positive nitrogen purge must be kept through the catalyst. It is wise to minimise the flow of nitrogen, even if it contains only a few ppm of oxygen, to avoid re-oxidising the catalyst.

When the loop pressure is reduced there is a possibility that water or steam may enter the synthesis loop due to any leaking heat exchangers. Special care is required under such circumstances, and if there is any doubt the water side of these heat exchangers should be de-pressured.

Re-oxidation will lead to a temperature rise and it is good practice to check bed temperatures every hour or so. Bed temperatures should be checked more often when work which can lead to oxygen ingress is in progress.

Unloading

Ammonia synthesis catalyst must be handled with great care when in the reduced state. In extreme circumstances, oxidation of ammonia synthesis catalyst can generate an exotherm of up to 1,600°C. Air must not be allowed to flow through reduced catalyst (e.g. by transporting in open containers) as this can create a chimney effect and draw more air into the catalyst.

Stabilisation is best carried out by slow oxidation. This can be achieved by keeping the catalyst wet and exposing it to air (over several days) outside of the vessel. The catalyst should be kept in a thin layer between 150–300 mm (6–12 inches) deep to allow it to cool easily. Self-heating can occur if the catalyst is dumped in heaps. Hydrogen is evolved by the reaction with water and the area should be well ventilated. Sources of ignition in and around this area should be identified and eliminated to prevent the evolved hydrogen from igniting.

Simple vessels are usually discharged by sucking out the material under nitrogen to prevent excessive oxidation. Air can be drawn into the top of the vessel and through the top part of the catalyst, but with sufficient nitrogen flow upwards through the remaining catalyst the quantity of hot material can be minimised, and trouble is unlikely. Nevertheless, temperatures must be watched carefully and if they become too high, the sucking must be stopped and the nitrogen blanket re-established.

Ammonia synthesis catalyst can be stabilised before discharge by circulating nitrogen with a suitable compressor and carefully introducing air. Complete re-oxidation is not necessary, and it is best to keep the catalyst temperatures between about 70 and 100°C. Even when stabilisation

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has been carried out it is advisable to keep a nitrogen supply available and to monitor bed temperatures carefully, as there is a possibility that some pockets of the catalyst will not have been stabilised and if a hot spot develops it can spread quickly.

Another method of stabilisation is to fill the ammonia catalyst basket with demineralised water. Demineralised water is used because converter internals are usually made of stainless steel. Since hydrogen will be slowly evolved, care must be taken to avoid accumulation creating an explosion hazard. Not all ammonia synthesis cartridges are suitable for filling with water. They may not be strong enough or they may contain insulating material, which should not be wetted. This feature should be checked with the manufacturer before the procedure is agreed.

It is not recommended to use steam in place of water to stabilise the catalyst as the hydrogen production is more rapid and effective stabilisation is not achieved.

Another point to note is that any unreduced iron left within the converter can potentially react with air to produce an oxygen deficient atmosphere and this possibility should be addressed as part of the confined space entry procedures and precautions.

Case studies

Catalyst loading into a Casale revamped reactor

Two converters on a Kellogg ammonia plant, were revamped with Casale internals. KATALCO 74-1R pre-reduced ammonia synthesis catalyst was selected for use in all three of the beds in both converters.

JM not only supplied the catalyst and the technical support services during catalyst reduction, but also arranged for inspection of the baskets during their fabrication and acted as a validator for the project.

To unload the previously installed catalyst, the converter was filled from the bottom with water to oxidise the catalyst. After the catalyst had been flooded for 36 hours, the basket was removed, and the oxidation of the catalyst began. The catalyst remained stable, but after two hours it caught fire due to high wind. After containing the fire, the catalyst was emptied into bins where it again began to glow after coming into contact with air.

Although the previous charge of catalyst had been flooded and no longer appeared active, there was enough residual activity to present a fire risk on exposure to air.

In preparation for loading, initially job methods called for the charge to be sieved, but as no dust was collected, job methods were revised to those suggested by JM. KATALCO 74-1R is considered suitable for use direct from the drum normally with no need to screen sieve.

The loading was started under dehumidified air. Due to hot work on the baskets, and the flammable nature of the rope ladder used in the converter, there was a fire during the loading. The air flow was stopped and nitrogen flow was started from the bottom of the converter. Following this, the rope ladder was replaced with an aluminium ladder. Measurement of the catalyst temperature showed that the catalyst had reached 160°C, and therefore work was stopped and the catalyst cooled under a nitrogen purge.

Contributory factors to the incident:

- damage to the passivation layer of the pre-reduced catalyst can result in oxidation exotherms, the unnecessary sieving of KATALCO 74-1R;
- lack of isolation leading to a chimney effect in the converter;
- flammable materials should not be present inside the converter during loading.

Following the rapid reduction of the KATALCO 74-1R charge, the converter effluent was vented to ensure that there was no residual dust that might damage the compressor. No dust was observed in the venting gas.

The customer was extremely happy with Casale and Johnson Matthey for the workmanship, product quality and technical support services during the operation.

Remote support for catalyst loading and reduction into a Topsoe converter

A new S-50 ammonia converter was installed during a plant turnaround in July 2020. The catalyst for the new converter was KATALCO 35-4 supplied by Johnson Matthey.

Since it was not possible to travel to customer sites due to Covid-19 travel restrictions in July 2020, JM were able to provide remote support to the customer in the loading and delivery of catalyst.

The quantity of catalyst supplied by JM was calculated based on the information provided during the tender. However, since there was information lacking regarding the dimensions and the exact volume of the catalyst beds, the supplied volume was approximately 20 m³ less than the volume available to fill.

Since the new converter was a radial bed, JM emphasised the point that it is very important that the catalyst bed is completely filled up to the bed covers and that it must be filled in such a way that loaded catalyst density for the entire bed is consistent and meets the target density to avoid voids in the catalyst bed and insufficient contact time for the required ammonia make.

JM recommended that the catalyst bed must be fully loaded with catalyst up to the cover plates and to the target density. Since additional catalyst needed to be procured to fill the bed as soon as possible, JM was able to offer available UK stock of KATALCO 35-4A for either sea freight or airfreight for delivery.

As part of the remote support for the loading, JM provided a catalyst operating manual, loading manual and log-sheets to the customer and offered advice on loading methods and vibration methods to ensure the correct loaded density of the catalyst.

JM also provided remote support to the reduction of the KATALCO 35-4 catalyst. Guidelines for the reduction of the catalyst were provided and a dialogue maintained between JM and site personnel. JM offered recommendations on reduction temperatures and reviewed reduction log sheets to assess the progress of the reduction.

Following completion of the reduction, the S-50 converter containing KATALCO 35-4 has operated successfully.

Catalyst loading into a KBR horizontal converter

In this case, the loading of the ammonia synthesis converter was to be carried out by a EPC Engineering crew under the guidance of both EPC and KBR engineers. Johnson Matthey was requested to provide additional support and was present for catalyst loading activities and training to ensure JM's loading standards were achieved. The loading was monitored by JM, KBR and EPC and inspections were carried out after each distinct layer was loaded.

Before the loading commenced, all pre-loading checks and requirements were completed. The vessels were inspected, the internal dimensions were checked against the mechanical drawings and the bed dimensions used for the loading calculations were confirmed to be accurate. The


PHOTOS: JOHNSON MATTHEY

Fig. 5: Loading of tkIS uhde process ammonia converters.

bed mechanical integrity was confirmed and the clearance to load was issued following the initial level markings.

The KATALCO series catalysts were inspected prior to loading and found to be in exceptional condition with no particles outside of the size range and very little dust. The drums were periodically inspected by JM and KBR to ensure the quality of the catalyst was maintained and all drums were found to be in very good condition.

The KATALCO 35-4 series catalyst was transported to site and kept in a designated shelter area close to the loading platform. From the shelter area the drums were lifted to the loading platform. All drums were always kept shaded in between layer loadings.

The KATALCO 35-8A and KATALCO 35-8C catalysts were supplied as pre-reduced and stabilised metallic iron. A meeting was held between JM, KBR, EPC and the site safety team regarding the loading of the pre-reduced catalyst KATALCO 35-8 in the ammonia synthesis converter. Although Johnson Matthey pre-reduced catalysts are specially designed to be stable when loaded in air, KBR specify in their loading documents that loading of pre-reduced catalyst must be done under nitrogen purge. JM supported KBR's decision to load the catalyst under a nitrogen blanket.

With expertise in the loading of catalyst in a nitrogen or oxygen deficient atmosphere, JM provided training and advice and recommended the use of breathing apparatus (BA) to eliminate the risk of a compromised atmosphere in a confined space. Detailed loading procedures and safety recommendations were communicated clearly to all participants.

Prior to commencing the loading, the nitrogen source for maintaining the inert atmosphere was identified and checked to be secure and reliable. All other nitrogen consuming activities were considered to ensure that adequate supply to the vessel blanket could be maintained.

Access and egress routes from the converter were assessed and established and the area around the converter was designated an exclusion zone with hazard barricading and signage placed around one metre m from the top cover and the base of the scaffolding. Atmosphere checks were carried out on a routine basis and multiple bed temperature measurements were taken and constantly monitored.

This extra precaution allowed the Bed 1 catalyst to be loaded in less than 12 working hours, without any temperature build-up or safety issues.

The loaded bulk density of the catalyst was checked after the loading and vibration of each distinct layer by the JM/KBR/EPC supervisor in order to ensure that the required target density had been achieved. All beds successfully achieved the target densities to within 1% of the target and the loading was confirmed to be very successful.

A close working relationship between JM, KBR and EPC and the site management team ensured a well-managed, safe and successful loading of the new build plant.

Loading catalyst into tkIS uhde® ammonia converters

Traditional methods of loading ammonia synthesis converters often included a step in which vibration is carried out across the bed to increase the catalyst packing density loading. Typically, after a 30 cm layer of catalyst was loaded a template was used to guide a pneumatic vibration device in repeatedly settling the catalyst, it is a discontinuous process.

Working jointly with tkIS in seeking loading improvements for use in uhde ammonia converters, JM developed the concept of a special conical distribution head to ensure even ammonia synthesis catalyst loading (Fig. 5). The design was checked and improved using DEM (discrete element modelling) to optimise the precise shape of the device and ensure it worked at a loading rate as fast as possible.

The device was tested in a full-scale testing rig at JM laboratories in the UK and it was seen to achieve loading densities matching those of vibrated catalyst loading, with very uniform spreading being witnessed in which the momentum of the catalyst was used continuously to settle both KATALCO 35-8 and KATALCO 74-1R pre-reduced ammonia synthesis catalysts to the desired loading densities.

The testing also confirmed that multiple loading heads could be used to increase the converter loading rate, the resulting loading process takes only approximately a tenth of the time versus traditional methods. The continuous loading process also leads to safer jobsite activities with less direct personnel exposure to catalyst, as the loading can be managed externally from the top and reduces the need for personnel to enter the confined bed space in the converter basket as it is loaded.

The loading method has been successfully used in all of tkIS uhde® ammonia converter designs loading either KATALCO 35-8 or KATALCO 74-1R pre-reduced ammonia synthesis catalysts and includes the world's largest plants operating reliably at 3,300 to 3,670 t/d.

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CLARIANT

ActiSafE™: The next level of safe ammonia synthesis catalyst reduction

M. Müller and S. Osborne

Clariant has developed a safe, efficient, and accurate method for measuring both water vapour formation and ammonia concentration during ammonia synthesis catalyst activation. ActiSafE technology measures water vapour formation at the outlet of the converter during ammonia catalyst activation and provides several advantages such as continuous real-time measurements of the water vapour and ammonia concentration. The real-time data allows plant operators to respond quickly to changes in water vapour formation and to optimise the reduction procedure, saving time, avoiding catalyst poisoning, obtaining a longer catalyst lifetime, and higher ammonia production.

Ammonia synthesis catalyst is delivered as iron oxide and the activation of the catalyst consists of reduction of iron oxide to elemental iron, using hydrogen in the synthesis gas. During the reduction, oxygen adhering to the iron crystallites reacts with hydrogen and forms water vapour, which is separated by condensation in downstream processes.

The reduction does not produce nor consume heat in any noticeable amount. However, heat must be supplied to reach catalyst temperatures where the catalyst is reduced. It should be mentioned the faster the catalyst heating rate, the higher the formation of water vapour.

The water vapour formed is a poison for the synthesis catalyst and has a direct impact on the formation of active sites; therefore, the formation of water vapour must be limited to certain concentration levels during the reduction. The main control criteria for the reduction of ammonia synthesis catalyst is a steady gas flow with a steady heat input, giving a well-controlled, moderate temperature increase in the catalyst bed(s) to keep exit water vapour levels within the desired limit.

Controlling the water vapour produced via the catalyst heating rate is therefore extremely important during the reduction. High heating rates can lead to excessive water generation, resulting in low catalyst activity, while too low a heating rate might cause a delay in production and lost revenue. Therefore, an accurate and continuous measurement of water vapour formation during the catalyst activation is crucial.

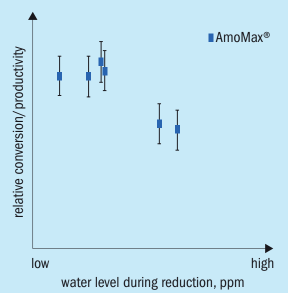
By using ActiSafE, a non-dispersive infrared (NDIR) based technology, the formation of water vapour can be monitored continuously, so the speed of reduction can be controlled and optimised to obtain the highest catalyst activity, fastest activation time, shortest time to production, and thereby better profitability of the plant.

Impact of water vapour formation on catalyst activity

Proper activation of the ammonia synthesis catalyst is very important, mainly because the pore structure is developed during the reduction. The catalyst pore structure is a very important parameter for catalyst activity because it facilitates gas access to the active sites. A slow reduction provides a fine pore structure and a large surface area in addition to a maximum number of active sites, hence ensuring the highest possible catalyst activity.

The formation of water vapour as a product of reduction has a negative impact on the formation of the pore structure and will poison the catalyst active sites. Furthermore, too high a level of water vapour will inhibit the ammonia reaction, which is exothermic, and the heat of this reaction is needed to assist in faster heat-up of the downstream catalyst beds since total heat input is usually limited by the capacity of the start-up heater.

Fig. 1: Impact of water concentration on catalyst performance



Source: Clariant

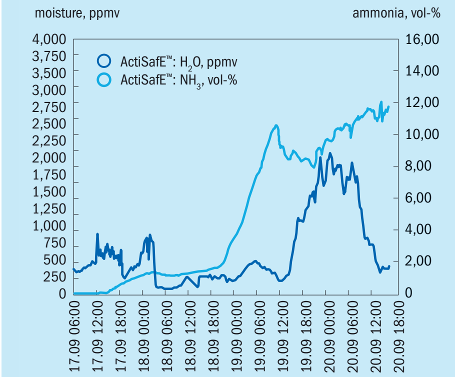
Clariant has carried out multiple investigations to assess the impact of water vapour formation on catalyst activity during the reduction. Fig. 1 illustrates that maximum catalyst activity (this is reflected by ammonia concentration at the outlet) is obtained at low water formation during the reduction. In this experiment, water formation was adjusted by varying the flow rates, while maintaining constant heat input and pressure. A low flow rate results in a high water concentration and vice versa.

Conventional method for measuring water vs ActiSafE

Due to the adverse effects of water, it is important to be able to determine the water content at the outlet of the converter correctly. Currently, three analytical methods are widely used to determine the water vapour formation at the outlet of the converter during the reduction. These commonly used methods are known as Ascarite, Carbide, and Karl Fischer titration method. All three methods have some operational challenges, such as:

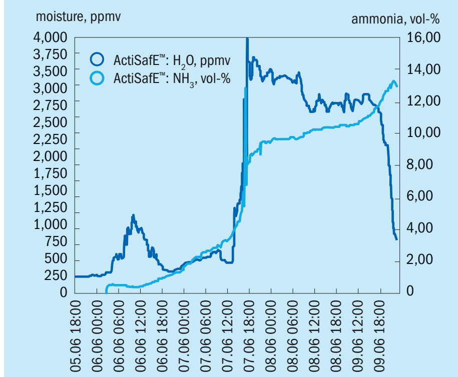
- Human errors must be avoided – the accuracy of the method requires highly experienced laboratory technicians.
- The Ascarite and Karl Fischer titration methods are far too cumbersome to be used by ordinary laboratory technicians. These methods are very time consuming and there is a delay before information about the water content of the sample is provided (the result of each reading may take three to four hours).
- All of these methods require a lot of chemicals and a high number of technicians.
- Condensation in the sampling line may not be detected on time, giving an error reading.
- Due to the discontinuous nature of the measurements (time delay), the speed of catalyst activation is normally slow, which could cause a delay in production costing a lot of money. If the water formation exceeds the limit, the ammonia reaction can be inhibited and will prolong the activation. For example, one-day production loss can cost $700,000 for a 2,000 t/d ammonia plant (assuming an ammonia price of 350 $/t).
- The time delay of the measurements prevents plant operators from being able to counter any sudden increase in water concentration (e.g. by reducing the temperature) before permanent damage is caused to the catalyst.

Fig. 2: Reduction of a 950 t/d plant



Source: Clariant

Fig. 3: Reduction of a 850 t/d plant



Source: Clariant

In contrast, the ActiSafE method developed by Clariant provides several advantages, which include:

- continuous real-time measurements of the water concentration in a range of 100-6,000 ppm, while also measuring the ammonia concentration at the same time;
- DCS system connectivity;
- there is no manual operation requirement as the instrument has an analog output and can be connected to the DCS system of the plant for monitoring the real-time values in the control room;
- quick reaction time;
- real-time data allows plant operators to react quickly to changes in water content and optimise the reduction procedure, thus saving time and avoiding catalyst poisoning, resulting in a longer catalyst lifetime and higher ammonia production;
- faster and safer reduction;
- by using ActiSafE, the reduction itself can be sped up within a safe range to obtain the highest possible activity without any risk of production delay;
- high activity and optimised operation conditions;
- measuring ammonia concentration at the converter outlet enables catalyst performance to be optimised by adjusting the bed temperature, hence ActiSafE will ensure high catalyst activity and optimise catalyst operating conditions to maximise plant profitability.

ActiSafE demonstration

ActiSafE has delivered on its promises when it has been tested in the field. During the field tests, the accuracy, reliability, and practicality of ActiSafE have been evaluated.

ActiSafE equipment consists of a single benchtop device, providing easy installation and use. It only needs to be supplied with electricity and lined up to the sample line, where care should be taken to avoid condensation. For optimum utilisation, the device should be connected to the DCS via the analog interface, providing real-time data in the control room.

Real-time measurement of the water vapour formation at the outlet of the converter enables plant operators to increase the reduction speed close to the highest possible level and to react immediately if this is required. The reduction can be performed in a safe, fast and accurate way in all cases. Monitoring the ammonia content gives information about the catalytic performance of the already reduced bed and the generated heat, which is in most cases essential for heating the beds downstream.

Case studies

The success and convenience of ActiSafE has been demonstrated at several reductions at a client site. Fig. 2 illustrates the reduction of a 950 t/d plant in Europe in 2018, where the temporary failure of the start-up heater was a particular challenge. With the help of ActiSafE the reduction was finished within the timeframe. The plant production above design capacity demonstrates the high quality of Clariant's AmoMax catalyst and the proper reduction.

ActiSafE showed reliable results during synthesis catalyst activation and was able to optimise and speed up the activation process without stressing the catalyst by forming too much water vapour. The information was also available during process disturbances, at any time of the day, without time delay and without the need for additional laboratory staff. In addtion, instantaneous concentrations rather than averaged values are measured giving a more realistic picture of the situation when compared with other techniques such as the Ascarite, Carbide or Karl Fischer titration methods. Having a more realistic picture increases the confidence of the operator who has full control over the reduction procedure. This is crucial as ammonia synthesis catalyst reductions are quite rare events for the production site.

Fig. 3 shows the reduction of two catalyst beds in a 850 t/d plant in Europe in 2019. It exemplifies how the reduction process can be accelerated with ActiSafE. For each bed the water vapour content was kept close to the upper limit which ensured a high reduction speed throughout the activation. During regular plant operation, the converter pressure could be decreased while keeping the production rate above design capacity. ■

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CASALE

Catalyst dense loading using Casale tools

F. Formentini

PHOTOS: CASALE

Above left: Fig. 1: Casale dense loading tool. Above right: Fig. 2: Detail of the fixed hopper on the converter top with details of valves for catalyst flow adjustment and hoses for catalyst feed.

The necessity to maximise catalyst load quantities together with the requirement to achieve a uniform catalyst density across the catalytic bed led Casale to develop its own dense loading procedure based on the use of Casale loading tools.

Casale loading tools have been widely used for catalyst loading in Casale ammonia converters since the 1990s. Compared to different commercial dense loading systems now in use with catalyst contractors, Casale loading tools (Fig. 1) are simple, have no moving parts, and can be fabricated easily by customers on site. Several tools sizes are available. Selection of the type and number of tools to be used is tailored by Casale for each project, based on experience, to get the best catalyst loading results. Casale typically specifies the type and number of tools required in order to provide a total loading speed of about 1 m^3/h.

The Casale dense loading system comprises a fixed hopper placed on the top of the converter, valves, plastic hoses, Casale loading tools and a frame to support the tools.

Loading speed is regulated using valves corresponding to the fixed hopper. The catalyst in the plastic pipes connecting the hopper and the loading tools is always free falling.

One operator is required to stay inside the converter in the upper part to operate the tools and to provide even distribution of the catalyst, while the catalytic bed is being loaded (Fig. 4).

Even though human presence is not required in other automated loading systems, Casale considers it an advantage to have an operator on site to control the loading process and avoid undesired situations that could occur when there is no physical monitoring of the job (maldistribution, sealing failures, catalyst presence in gas areas).

Pre-reduced catalyst is required to be loaded in cool and dry air, but with all the provisions to swap to a nitrogen atmosphere if a rise of catalyst temperature is detected.

Temporary thermocouples are used to monitor the temperature of the pre-reduced catalyst during loading.

The catalyst falls in plastic hoses to the tool, where it impacts a cone which acts like a sprinkler, evenly distributing the catalyst in all directions (Fig. 3). The catalyst falls from the top of the catalytic bed to the bottom. This system has been used with good results for a maximum free-falling height of about 12 m for oxidised catalyst and about 10 m for pre-reduced catalyst.

A loading operator is not required to access the catalytic bed, because no vibration or raking is required, making this loading system safe and suitable even for very slim catalytic beds, where human access is not possible.

The Casale dense loading system results in a very high and uniform catalyst density throughout the bed height, an important factor in obtaining the best performance from the converters.

Catalyst unloading

Casale has participated in the unloading of hundreds of catalyst charges from ammonia converters during its long history of supplying and revamping ammonia converters and has developed its own bed design to facilitate this operation that can sometimes be quite difficult and time consuming.

Ammonia synthesis catalyst is usually unloaded via vacuum in a nitrogen atmosphere. Nitrogen vacuumed with the catalyst is recycled and sent back to converter after cooling to reduce the nitrogen costs.

Catalyst passivation for further unloading in air is strongly discouraged, as steam flow control in the gas going through the catalyst is a difficult task. Major damage to the converter internals, and even melting of the steel components have been reported in the past.

To avoid catalyst vacuuming, the open-top ammonia converter designed by Casale can also be unloaded via a drop-out nozzle. This feature was introduced about 15 years ago, and was originally designed for slim converters where operator access for vacuum purposes was not possible and unloading by basket tilting was not a preferred option.

PHOTOS: CASALE

Fig. 3: Casale dense loading tool spreading the catalyst in the bed.

Fig. 4: Catalyst loading operator inside the cartridge during loading work.

PHOTO: CASALE

Fig. 5: Lifting of basket full of active catalyst purged with nitrogen – plastic wrapping to avoid the catalyst coming into contact with oxygen.

PHOTOS: CASALE

Above left: Fig. 6: Lifting of the basket full of active catalyst purged with nitrogen.
Above right: Fig. 7: Catalyst unloading via a drop-out nozzle.

The catalytic bed full of catalyst is designed to be lifted out of the cartridge (Fig. 6) and is easily unloaded by opening the drop-out nozzles provided on the bottom of the catalytic bed.

The catalytic bed, continuously purged by a nitrogen source feeding a sparger pipe, is wrapped in plastic during its removal from the cartridge to avoid oxygen coming into contact with the active catalyst and consequently raising its temperature.

The basket is rested on a proper structure and the drop-out nozzles (two or three) are opened one by one, dumping the catalyst into dedicated skips (Fig. 7).

The use of drop-out nozzles requires heavier liftings to be executed for the removal of baskets full of catalyst, but greatly reduces the time required for catalyst unloading work, as well as reducing the time spent by personnel working in a confined space with a life support system.

The nitrogen quantities required during unloading are also significantly reduced when using the drop-out nozzles for unloading.

Casale designs baskets that can be lifted full of catalyst for small to medium catalyst volumes of up to about 15 m^3. For bigger volumes, a design using special lifting devices can be applied.

An additional advantage of baskets that can be lifted full of catalyst is that they can be pre-loaded outside of the converter, and in parallel, before their installation, reducing the time required for catalyst loading activities and improving the safety of this operation. ■

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31

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HALDOR TOPSOE A/S

A safe solution for pre-reduced ammonia synthesis catalyst replacement

C. A. Diaz-Tovar and B. Hartvigsen

Nowadays, a catalyst is state-of-the-art when it not only delivers superior operational reliability but also logistics strength. These features are of particular importance as the catalyst could reach an operational lifetime as long as 30 years, making the installation and activation of the new charge (i.e. replacement) possibly a once-in-a-lifetime experience for most of the personnel in an ammonia plant.

The pre-reduced Topsoe ammonia synthesis catalysts KMR 111 and KM1R are manufactured to deliver high activity during normal operation as well as to minimise the risks associated with handling and loading, while also ensuring a shorter start-up period.

The unloading, installation/loading and activation/start-up need to be meticulously planned to avoid any incidents, which could result in serious personnel injuries and/or equipment damage, while minimising plant down-time.

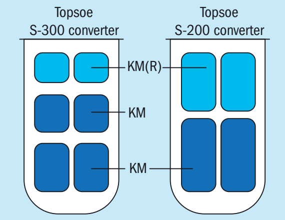
In general, any catalyst installation is associated with many hazardous activities. Among this, the confined space entry in a long and narrow reactor with limited egress is one of the most challenging from a safety standpoint; especially when the catalyst to be loaded is in its pre-reduced state as it is liable to self-heat in contact with air and without an energy supply. Furthermore, when this type of catalyst is not passivated to withstand the weather and work conditions found in a typical loading, the installation normally takes place under an inert atmosphere.

Consequently, having a stable ammonia synthesis catalyst in its pre-reduced state that can be installed in a normal atmosphere is highly valuable for any ammonia plant owner from a safety and economic standpoint.

Although installing a pre-reduced catalyst has the drawbacks previously mentioned, they are significantly outweighed by the incentive of reducing by two to four days the time that the plant is operating in transient conditions, i.e. activation/start-up of the ammonia synthesis catalyst.

The benefits that the pre-reduced version of Topsoe KM catalysts have brought to the ammonia industry are tangible. In more than 1,200 charges of ammonia synthesis catalyst that have been successfully

Fig. 1: Recommended loading scheme for ammonia synthesis converters. (R) denotes prereduced catalyst



Source: Topsoe

manufactured, installed, and activated, the vast majority have been loaded with Topsoe's recommended combination of pre-reduced and unreduced catalysts (Fig. 1).

The path to highly stable pre-reduced ammonia synthesis catalysts

Clearly, the quality of the raw materials, the optimal amount of the promoters and the manufacturing procedure, are all key factors that determine the final quality of an ammonia synthesis catalyst. However, to produce a catalyst that offers both operational reliability and logistics strength, the path from production to normal operation must be carefully planned.

The steps that allow the production of a highly active pre-reduced KM catalyst that is safe to handle, transport, install and activate are discussed below.

Reduction

The two main factors that influence the intrinsic catalyst activity are the reduction procedure, specifically how the procedure is designed to limit the effect of water, and the particle size in which the catalyst is reduced from iron oxide to elemental iron.

The effect of water

Water and high temperatures are promoters of hydrothermal sintering. This phenomenon is characterised by the growth of iron crystallites, which has a negative impact on the catalyst activity as it decreases the available surface area and could also block or collapse pores, making the crystallites inaccessible to the reactants.

Additionally, water inhibits the ammonia synthesis reaction; which means that higher temperatures are required for reaction to take place.

Particle size

The larger the particle size, the longer it takes to complete reduction of a given particle and the more damage is therefore inflicted to the following catalyst layer due to water released in the reduction of this particle.

Reduction procedure

The reduction method influences the properties of ammonia synthesis catalysts.

The reduction procedure should ensure that the pore system, formed by the renewal of oxygen ions, increases the specific surface area of the catalyst as close as possible to the theoretical maximum. The characteristics of the pore structure that develop during the reduction also depend strongly on the heating rate and the gas composition (mainly H_2O) to which the catalyst is exposed.

A controlled heating rate is important for the activity of the catalyst. It prevents rapid reduction, which leads to high water vapour concentrations in the gas that accelerates changes to the crystalline structure of the already reduced catalyst.

It is therefore of utmost importance to limit the amount of water vapour in contact with the catalyst and this can be achieved with a high space velocity. Although, an initial space velocity of 3,000-5,000 V/V/H is difficult to achieve in an industrial installation, it can be done in the manufacturing facilities of the KM catalysts.

A pre-reduced catalyst charge will then not only ensure a higher activity of the catalyst but also that the heat generated by the ammonia synthesis reaction will allow the plant to increase the space velocity and to keep it as high as possible throughout the activation of the charge. This will lead to a more controlled heating rate and a lower water vapour concentration in the exposed catalyst.

Passivation

A reduced ammonia catalyst is strongly self-heating when in contact with air even at ambient temperature. Consequently, the pre-reduced catalysts used in the industry have been given a passivation treatment upon reduction so that they can be transported and installed.

The passivation treatment is carefully conducted considering that:

- Skin oxidation has an influence on the pore structure and the surface area, since voids may be closed off and the mouth of the pores shrunk reduced.
- It is mandatory that KM-catalysts comply with the international transport regulations UN 2881 Metal Catalyst, Dry (Iron), Class 4.2, Packing Group II; which are part of the document "Recommendations on the Transportation of Dangerous Goods, Model Regulations".

Additionally, the state-of-the-art passivation treatment given at manufacturing facilities provides Topsoe's ammonia synthesis catalysts, KMR 111 and the industry-leading KM1R catalyst, with sufficient stability so that they can be loaded in atmospheric air.

Installation/loading

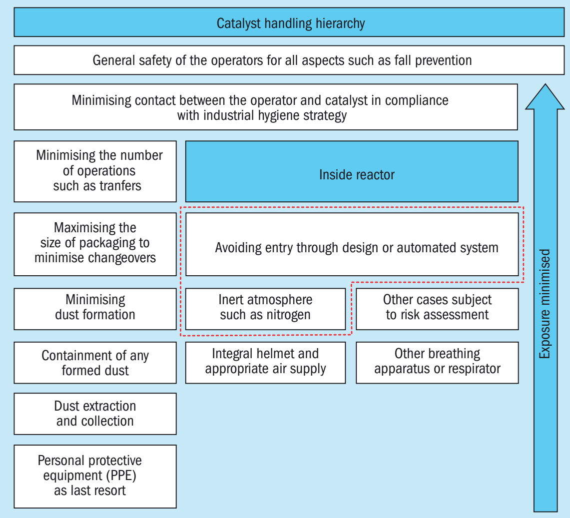
The loading of the catalyst is usually overlapped with the mechanical installation of the beds, inter-bed heat exchangers, among others, and it is associated with many high-risk activities such as: handling and exposure to hazardous substances, working at heights, and the most critical one, confined space entry in a long and narrow reactor with limited egress.

According to the catalyst handling hierarchy (see Fig. 2) issued by the European Catalyst Manufacturers Association, avoidance of reactor entry is the preferred option. However, if reactor entry is required, particularly in the case of inert/nitrogen atmosphere (or other potentially hazardous residual or incidental substances), an appropriate breathing apparatus such as an integral helmet and appropriate air supply is normally required.

Reactor inert entry is a high-risk operation and strict procedures are required including, in the case of European countries, Europe Conformity (CE) approved specialised equipment, supervision and training.

Over the past decade, at least two full charges of pre-reduced catalyst and over ten of the prereduced/unreduced combination

Fig. 2: Catalyst handling hierarchy



Source: Topsoe

have been installed in atmospheric air every year worldwide without any incidents, demonstrating that Topsoe pre-reduced KM catalysts possess a unique feature that allows customers to not only reduce the loading time, but also to avoid the additional safety risks and hazards associated with inert entry.

In addition, from an economic perspective the cost of loading a pre-reduced catalyst in an inert atmosphere can be at least 25% higher than loadings done in atmospheric air. These expenses are not insignificant when one takes into consideration that the cost of loading a standard size 80-m³ ammonia synthesis converter in a nitrogen atmosphere is approximately $200,000 (additional cost of one bed in a split charge is approximately $50,000).

Activation and start-up

The protective layer added during the passivation treatment must be removed again before the catalyst regains its activity for ammonia synthesis.

The amount of water formed during the re-reduction of the catalyst is much lower than the one formed during the reduction of the oxidised catalyst. While it is expected that the charge with only pre-reduced catalyst stops forming water as early as 36 hours after the activation has started, the full charge of unreduced catalyst will continue forming water during the first eight to ten days after the activation commenced.

However beneficial a much lower amount of water produced during the activation of the catalyst charge is, the biggest benefit of using pre-reduced catalyst is the possibility to reach design capacity within a few days.

In tests, the estimated production of ammonia during the activation of three 90 m³ catalyst charges with different content of pre-reduced catalyst (0, 30 and 100%) showed a significant increase in the production rate (twofold) when moving from a full unreduced charge to a split charge of pre-reduced (first bed) and unreduced (lower beds) catalysts.

However, this same step-up increase is not seen when moving from that same split charge to a full charge of pre-reduced catalyst. Due to this, the majority of the ammonia synthesis charges installed in the world are split loadings, but local circumstances, such as limitations on the disposal of water containing low concentrations of ammonia, can make a full pre-reduced loading optimal.

Topsoe is always ready to assist with finding the right loading scheme depending on requirements. ■

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Opportunities for data-driven analytics

As industry trends add pressure to optimise processes, new digital technologies for round-the-clock, data-driven decision-making can help plants boost production efficiency without time-consuming manual analysis or large-scale investment. Here, **Dr S. Werner** of Navigance explores their potential and shares the difference they are already making.

When Navigance conducted a recent study[1] of over 40 experts working in various chemical manufacturing roles, what they revealed about the key priorities in the sector and the role digital technologies can play in addressing them was fairly unanimous.

Most saw the potential in digitalising aspects of their operations and making better use of the data being collected across it. Many recognised that artificial intelligence and other advanced analytics technologies could help to unlock operational efficiencies by enabling more data-driven decision-making.

However, the extent to which they felt ready to take the steps needed to achieve that themselves varied greatly. This depended largely on the 'demographics' of their company: size, level of digital maturity, and how effectively they were dealing with the priorities most said came ahead of efficiency.

Most crucial was the need to ensure safety and minimise time lost to accidents. Next was keeping plant availability high, preventing downtime, production losses and their potentially high costs. Only once safety and availability were being managed at consistently high levels would plants turn their focus to increasing the efficiency of their processes.

Increasingly, though, manufacturers may not have the comfort of waiting "until the time is right" to optimise their processes. Trends gathering pace in the sector for some time look set to make optimisation a necessity rather than a nice-to-have.

For one thing, the effects of economic downturn in recent years mean the ability to do more with less – from people to energy to raw materials – has never been more pressing. The Covid-19 pandemic has exacerbated this trend, with over 32,000 production jobs lost in the sector in the 12 months from September 2019 alone[2].

Chemical producers are also struggling to replace lost experience as an ageing workforce retires, taking with them skills and a problem-solving intuition that are hard to replicate quickly enough in new recruits.

What's more, the coming years will see a keener focus on the sustainability of chemical processes. Rather than simply offsetting the impact of their operations, chemical manufacturers must radically rethink their ways of working and make much more efficient use of resources and materials.

The data dilemma

For those prepared to act now, digital technologies, big data and the expertise to harness them effectively can help to overcome these challenges and overtake those who continue to wrestle with them.

However, just 4% of people Bain & Company surveyed[3] from over 400 companies with $1 billion-plus turnovers said they had the resources needed to draw useful insights from and act on their data. Indeed, Navigance's own survey found the greatest barrier to digital adoption was plants' ability to spare time or people for complex manual data analysis or to implement technologies that would take that burden off their shoulders.

The good news is that it is possible to use plants' data to optimise both availability and process efficiency, without tying up in-house resources, big investments or even long periods before seeing the benefits. It can be done in a way that improves both the sustainability and profitability of operations while reducing production costs. And it builds on technologies and methodologies that are already in place.

The opportunity in chemicals

The chemical sector is in a good position to reap the benefits of data-driven decision-making.

Use of control systems has been widespread since the 1960s, and a typical plant now has thousands of sensors collecting huge volumes of process and performance data. This data is used to help control the plant, generate reporting and may also be stored long term to support troubleshooting and analytics.

Much of this work is done retrospectively and at irregular intervals, though, rather than routinely and in real time, when it can make the greatest potential difference. Also, significant amounts of that work are done in manual workflows, which take time and resources and are prone to errors. It also depends on plants being able to spare personnel for extended periods to conduct the necessary analysis. A challenge at the best of times, but even more so given current economic conditions.

Decisions based on this data, though grounded in many years of industry experience, are usually made using intuition and a 'gut feeling' more than an irrefutable reading of the facts. A skill hard to teach to a new generation who will carry the baton forward.

In addition, the sheer volume of data being analysed manually makes it highly likely that important patterns and potential will be missed; they simply can't all be spotted by human eyes. And if the data used is only ever historical, plant teams will always be on the back foot.

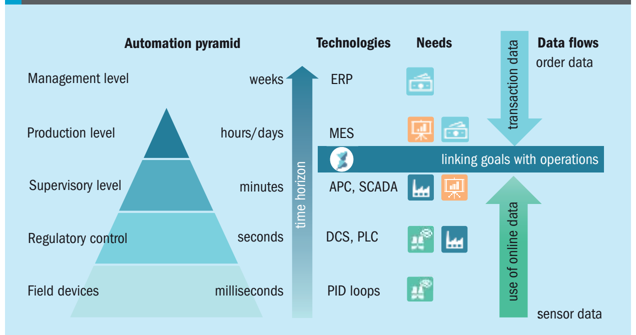
The opportunity digitalisation presents is to integrate continuous process data into operational decision-making. Moving from retrospective analysis and troubleshooting to a comprehensive and near real-time view of how the operation is running to inform confident, effective action.

Doing so can help to close the gap between commercial and operational goals.

The automation pyramid in Fig. 1 shows layers of decision-making from transactional data-based ERP system orders down to the stabilisation of individual measurements in PID loops. Each layer addresses needs, such as safety and availability in the bottom up to margin and efficiency above.

Navigance helps operational teams deliver on their commercial goals by providing expertise that is hard to acquire in house, and an approach that builds on and surpasses established methods of understanding and improving how plants work.

Fig. 1: The automation pyramid



Source: Navigance

The challenges of modelling

Models are a vital tool for the chemical sector and engineering sciences in general. In essence, they are mathematical representations of the relationships between variables in (real) systems. In the chemical industry or continuous manufacturing in general, they can describe and help operators understand the behaviour of a plant or process.

Models based on first principles have been used for decades to help design new plants or revamp, debottleneck, and optimise operations in existing ones. But modelling real-world scenarios in today's highly complex operations can be very challenging.

First principle models are based on established science and often explicit relationships in a particular unit operation in a chemical process. They rely heavily on recognised correlations and a fundamental understanding of underlying 'ab initio' physio-chemical phenomena such as kinetics or chemical equilibria or pure substances. However, on closer inspection that understanding is not really complete at all.

In many cases, a flow-sheet model combining first principles with correlations such as heat transfer, porosity and vapour pressure can be constructed to describe the full extent of the plant. Even getting vapour-liquid equilibria (VLE) data for a non-ideal multi-component mixture can be limited by available correlations or validity ranges. Understanding the pressure drop of irregular catalyst particles in a packed bed relies on estimation rather than on exact calculation. It can take many years of work, lots of resources and substantial investment to address these points.

In addition, observed 'real world' information is usually needed to "fill in the blanks" where parameters are unknown or missing. But getting it becomes increasingly difficult as the complexity and number of parameters involved increases.

In an attempt to describe the phenomena seen in real-world systems sufficiently, it is easy to end up with thousands of model equations and partial differential equations that cannot be solved analytically.

This is when "fiddle factors" come into play; widely established parameters used to fit model outputs to the observed data. This includes "activity factors" to correct for deviations in catalytic activity, or "shape factors" to account for inaccuracies in the pressure drop.

These factors compensate for unknowns or uncertainties in the models but can result in physically unrealistic situations in parts of the simulation and leave significant inaccuracies in the final result.

Describing a plant to its full extent can mean stacking many different first principle based models, and all their potential inaccuracies, on top of each other. Bridging this gap between model and the day-to-day reality in plants, takes expert care and a fresh approach that builds on and enhances what has gone before.

Data-driven hybrid models

Hybrid models can help overcome these gaps by harnessing the large data volumes already available from process control systems and data historians in place across the industry.

A hybrid approach provides flexibility to not only present a real-world view of plants but also adapt to changing conditions within them. It can also avoid the trap of "spurious correlations," which are often the result of a purely statistical approach and bring little value in optimisation[4].

The benefits of using data-driven neural networks and hybrid models to dynamically model chemical systems have been recognised for some time – described in academic contexts as much as 20 years ago[5]. Applying the theory in practice, however, involves obstacles it takes expert care to overcome.

As we have heard, the data required usually exists, but it needs preparing and processing in order to be useful. This can prove both time consuming and expensive if done manually. Data scientists spend up to 80% of their time preparing data for analysis, with the vast majority of that spent cleaning and organising the data[6]. Fortunately, the days of needing to do so are fading.

Progress in making access to data easier, combined with the decreased cost of computation and data storage, and the rapid advances seen in machine learning technology, now presents the opportunity to create data-driven models that build on automatic and continuous flows of high-quality, cleaned data for advanced and automatic analysis.

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The Navigance approach

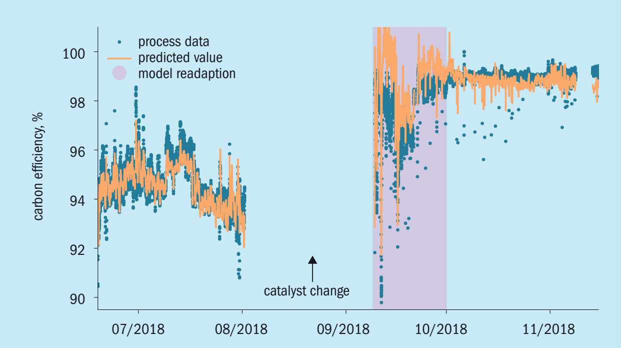
Over recent years, Navigance has developed and demonstrated the successful use of next-generation, data-driven hybrid process models that adapt easily to both a plant's historic and real-time data, generating reliable, prescriptive advice teams can use to continuously optimise their operations.

These models are still based on established first principle techniques, physico-chemical relationships and engineering principles. The difference is they are augmented with neural networks adapted to each plant's specific setup and circumstances, and machine learning algorithms that provide advanced and almost instantaneous analysis of the data.

In contrast to rigid first principle models, Navigance hybrid models learn intuitively and respond quickly to changing conditions and factors, from varying load scenarios to deactivating catalysts. And they work regardless of the plant's technologies and setup.

In the example shown in Fig. 2, Navigance models were tailored to suit a plant's specific process based on an initial data set as it introduced a new catalyst. It shows the quick adaptation of a machine learning model to a new situation in a plant. After a catalyst change the models give high-quality predictions quickly after the initial, unsteady start-up phase.

Fig. 2: Example of the quick adaptation of a machine learning model to a new situation in a plant



Source: Navigance

Real benefits, rapid results

With Navigance base models ready to adapt to each plant's particular setup and circumstances, they can be deployed quickly, and realise process optimisation potential faster.

Navigance works with producers to implement a detailed optimisation strategy tailored to their most pressing operational goals and any constraints and limitations. This includes identifying appropriate control variables with which to steer their operations and increase process efficiency.

In the usage phase, a continuous stream of plant data is uploaded securely to the Navigance cloud. There it is automatically cleaned and prepared to remove all outliers, noise and discrepancies using adaptive filters, pattern-matching heuristics and other additional classification techniques.

This cleaned data provides the foundation for automatic forecasting and real-time, prescriptive recommendations based on a complete and current view of the most pertinent information. Plant personnel can access this advice in an online dashboard and act on it with confidence to refine process control variables, hit their optimisation goals and critical KPIs, and so deliver on the plant's commercial objectives.

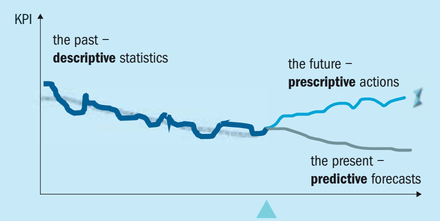
In this way, producers can take data-driven decision-making to new levels. Moving away from hours spent analysing and cleansing 'descriptive statistics' and beyond predictive forecasting to a future of prescriptive advice for round-the-clock optimisation (Fig. 3).

It's a future that producers of chemicals other than methanol are already seeing rapid benefits from today, too.

In formaldehyde production, for example, where methanol equates to over 90% of total production costs, the amount of product it yields is a vital KPI to measure and optimise.

Within just three months of introducing the Navigance Optimization Engine at one of its plants, one large formaldehyde producer with multiple units found the models and recommendations helped increase yield by 0.8 mol-% above the long-term average typically seen.

Fig. 3: Different time domains in data-driven analytics



Source: Navigance

Fig. 4: Fluctuation of methanol production yield

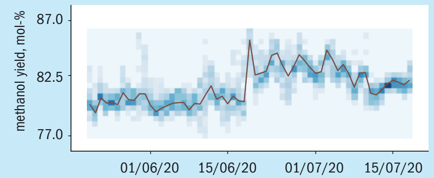


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Fig. 5: Assessment of occurrences in methanol yield over one week

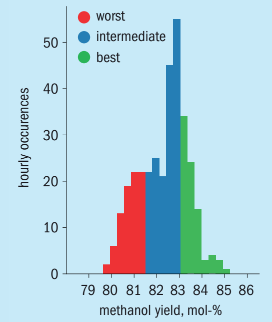

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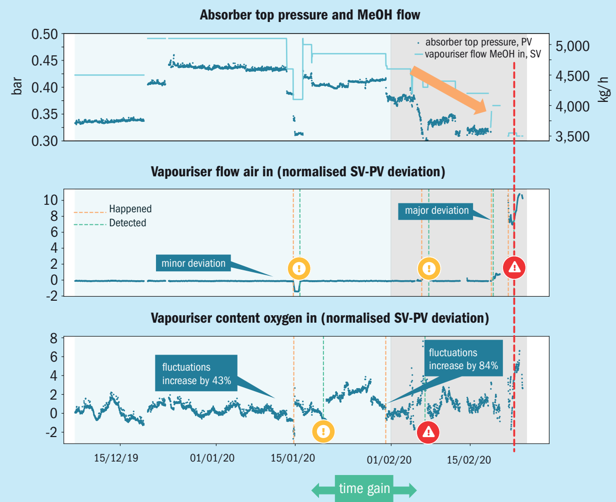
Fig. 6: Depiction of issues in different measurements identified and generated by algorithms before a shutdown



Source: Navigance

Assessing optimisation potential

A common question producers ask when deciding if and when to introduce data-driven optimisation is: "how much benefit can we expect in our plant?" One method Navigance applies to assess a plant's optimisation potential is the Best Demonstrated Practice (BDP) approach[7].

The underlying idea is that plants do not always run at their optimal level, so exhibit efficiency fluctuations in the trends of their major KPIs. In one example from a methanol plant, the KPI in question – the yield – can be seen fluctuating over time (Fig. 4).

By exploring how well the plant has proven to perform in a given week, then looking at hourly performance, it can be assumed that continuous optimisation and fine tuning of the process in a given week can lift the worst-performing tercile up to the average performance demonstrated in the same week. In the same example (Fig. 5), the weekly average optimisation potential ranged from a 0.2% to a 0.4% increase in yield.

This is only one aspect that is explored. Further optimisation potential can be found in cross correlations between variables and KPIs. Assessments of high-resolution plant data commonly reveal optimisation potential of 1-3% for a methanol or ammonia plant.

Plant monitoring: an additional benefit

Navigance hybrid models offer producers other optimisation potential too. A by-product of the data cleaning and preparation process is the ability to detect anomalies, unusual behaviour and potential issues early. This forms the basis of a solution called the Navigance Plant Monitor, which combines 24/7 monitoring of the plant with predictive alerting.

In the example shown here (Fig. 6), unusual behaviour detected by multiple separate algorithms highlighted the critical failure of a valve more than two weeks before it happened. This allowed maintenance teams to take proactive and focused action.

Conclusion

For chemical producers yet to take substantial steps into a digitalised future, or those eager to accelerate the process but struggling to spare the time needed to do so, these technologies offer a real opportunity.

Navigance offers solutions to help address both the industry's most critical priorities and the accelerating trends that look set to shape how it operates in the years to come.

Ready to tailor to each plant's specific setup or existing technology mix, and to implement and start using quickly without huge expense, these solutions are backed by the expertise in both chemical processes and data science needed to deploy them for maximum effectiveness. ■

References and notes

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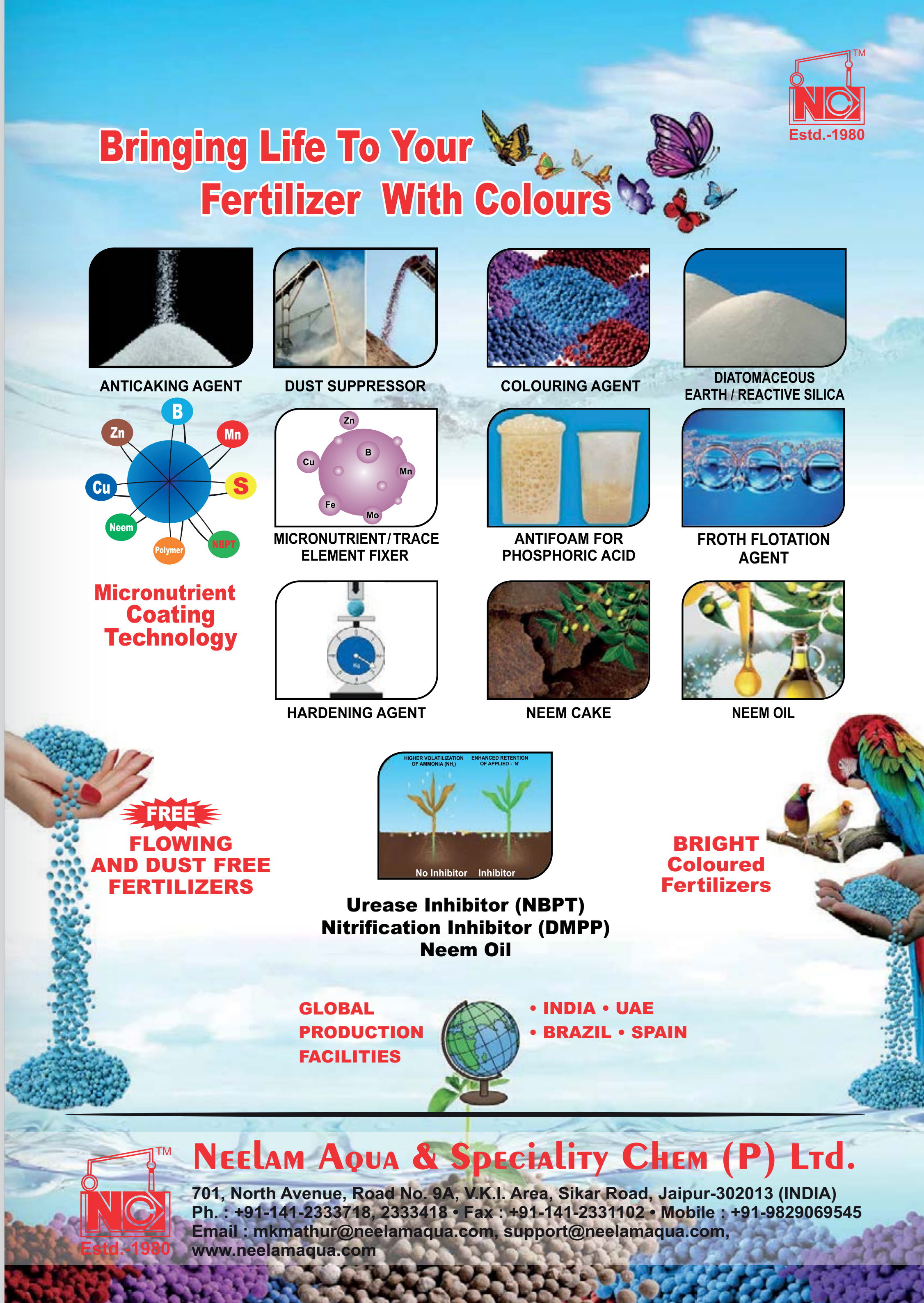

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