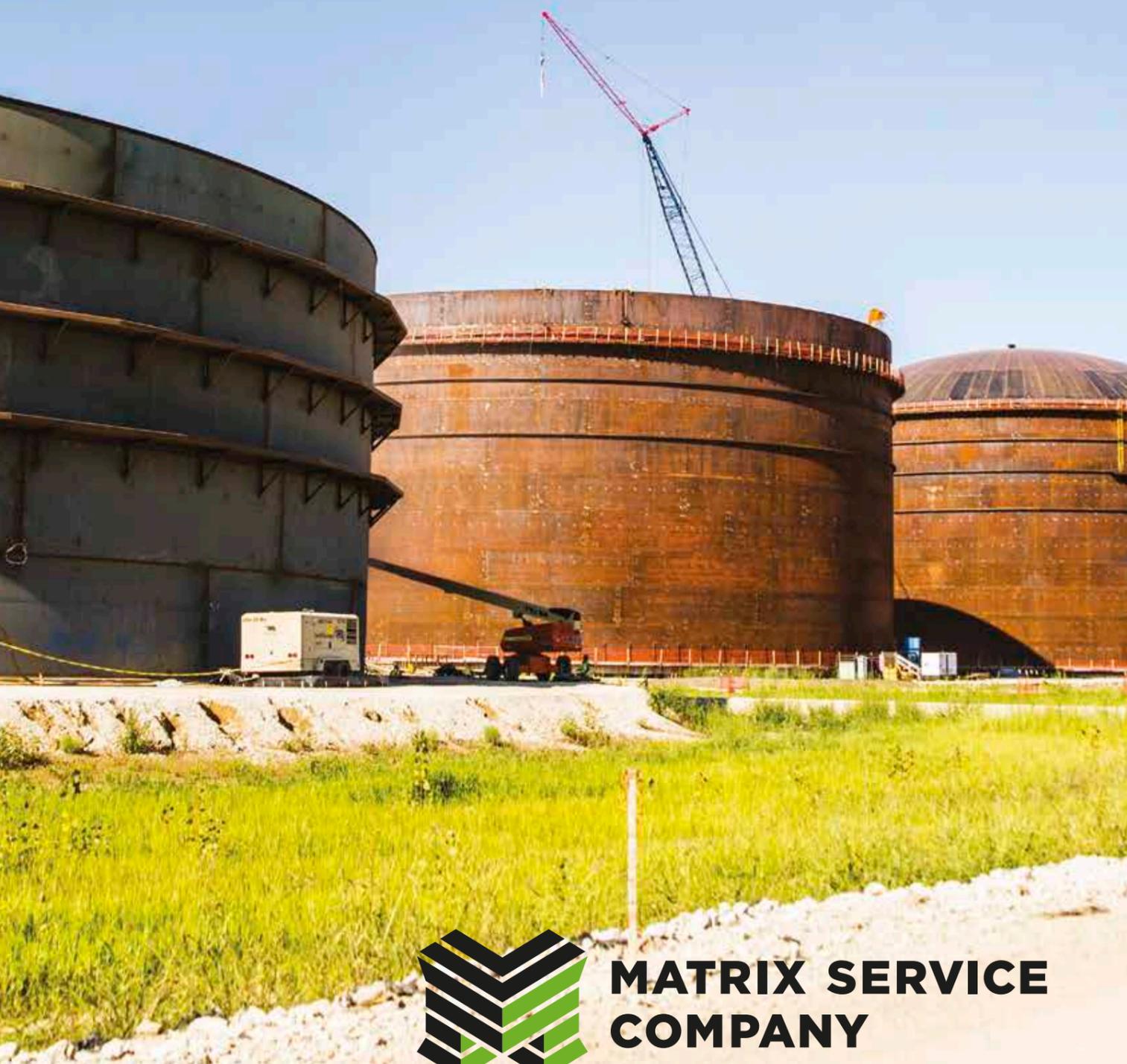


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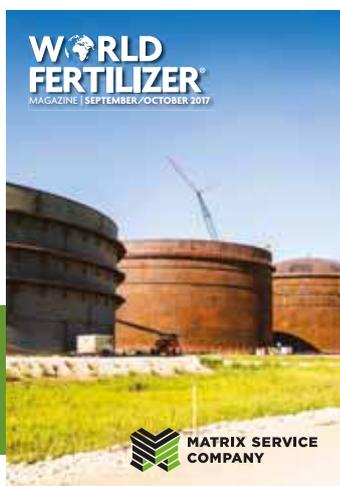
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Increased oil and gas production, as well as environmental regulatory demand for lower sulfur levels in transportation fuels, has created a need for efficient sulfur storage and transportation logistics. Industry leader, Matrix PDM Engineering, outlines the infrastructure considerations for doing so. For more information: www.matrixpdm.com



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COMMENT

CONTACT US

MANAGING EDITOR

James Little
james.little@worldfertilizer.com

ASSISTANT EDITOR

David Rowlands
david.rowlands@worldfertilizer.com

ADVERTISEMENT DIRECTOR

Rod Hardy
rod.hardy@worldfertilizer.com

ADVERTISEMENT MANAGER

Chris Atkin
chris.atkin@worldfertilizer.com

ADVERTISING EXECUTIVES

Will Powell
will.powell@worldfertilizer.com
David Ramsden
david.ramsden@worldfertilizer.com

PRODUCTION

Ben Munro
ben.munro@worldfertilizer.com

SUBSCRIPTIONS

Laura White
laura.white@worldfertilizer.com

WEBSITE MANAGER

Tom Fullerton
tom.fullerton@worldfertilizer.com

WEBSITE EDITOR

Callum O'Reilly
callum.oreilly@worldfertilizer.com

DIGITAL EDITORIAL ASSISTANT

Angharad Lock
angharad.lock@worldfertilizer.com

REPRINTS

reprints@worldfertilizer.com

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JAMES LITTLE, MANAGING EDITOR



Hurricanes, typhoons and tropical storms are a fact of life in many parts of the world and no less so than parts of the US and the Caribbean. Nonetheless, 2017 has been a particularly brutal year. Although the hurricane season is not officially over until the end of November, a series of category 3 or greater hurricanes have already churned through the region wreaking unprecedented havoc and destruction over a vast area. A number of Caribbean islands have been left almost entirely destroyed, without power and with many thousands of people displaced from their homes. The clean-up and rebuilding operation across the region will take years and with the majority of these islands

heavily reliant on the tourism industry, the fallout from the 2017 hurricane season will be severe.

The southern US states of Texas, Louisiana and Florida were hit hard and in quick succession by hurricanes Harvey and Irma with significant infrastructural damage and the loss of over 100 lives. For the US, 2017 ranks as the 17th deadliest hurricane season since 1900. The 2005 season, still fresh in the minds of many, which included Katrina and Rita, ranks in fifth place, whilst the most deadly season on record was in 1900 when the Galveston hurricane led to an estimated 8000 deaths.

It is clear that the damage and disruption caused by Harvey and Irma will have an impact on the energy and chemicals sectors in the US. Texas is home to 31% of US refining capacity and the Houston chemical complex accounts for almost two thirds of US primary plastics production and 45% of basic chemical production. Many facilities were inundated with floodwaters in the wake of record rainfalls and the media was full of images of submerged refineries and petrochemical plants. Arkema's Crosby plant grabbed the headlines with reports of its impending explosion due to floodwaters knocking out its primary and backup refrigeration systems, bringing back vivid memories of the 2013 West Fertilizer Company ammonia nitrate explosion. However, in the event, whilst the initial storm impacts were severe, the longer term effect on the energy and chemicals industry appears to be short lived. Refineries and chemical plants, including the worse hit such as the Motiva refinery in Port Arthur, are steadily coming back onstream. The Mosaic Company's phosphate facilities in Florida appeared initially to be the greatest concern within the fertilizer sector but the company was quickly able to report only limited damage.

It appears that a combination of improved weather forecasts from the US National Weather Service and lessons learned from previous hurricanes in the Gulf of Mexico have had a major impact on the energy and chemical sector's ability to prepare for disaster. The *Financial Times* reports Karen Harbert, President of the Global Energy Institute at the US Chamber of Commerce, as saying, "The industry learnt a lot from Rita and Katrina, and invested in resiliency. They hardened a lot of facilities to withstand wind and flood damage." Examples of this are widespread and include better backup systems, control rooms and substations relocated to higher ground and the construction of improved floodwater defences.

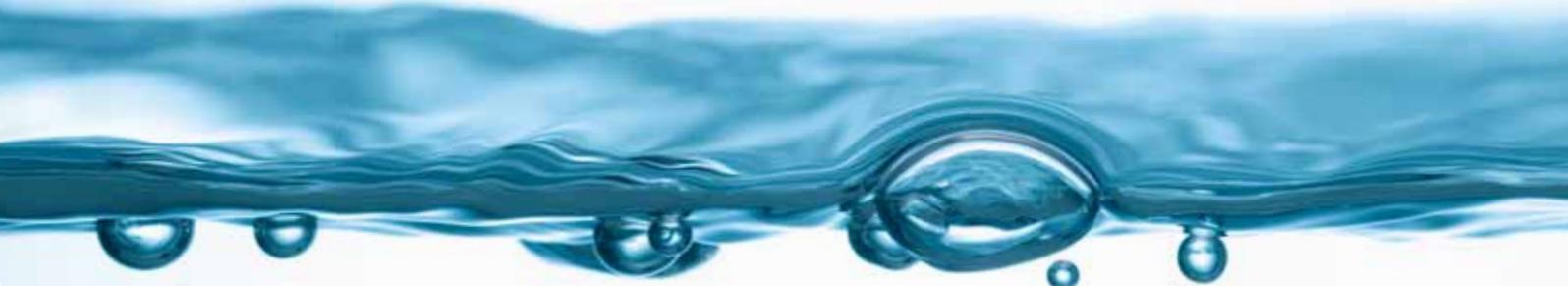
That these measures have had such an immediate impact is encouraging especially in the light of research into global warming and warnings of the increased threat of severe weather events escalating in years to come. Clearly there are lessons still to be learnt as the Arkema Crosby plant demonstrates, but that they are being heeded is good news indeed for this critical industrial sector located as it is in the hurricane belt.



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WORLD NEWS

BRUNEI thyssenkrupp awarded EPC contract for fertilizer plant

thyssenkrupp has received a new order from state-owned Brunei Fertilizer Industries for the engineering, procurement and construction (EPC) of a new fertilizer production facility. The order was signed on 26 August and is expected to come into effect later this year.

The new fully integrated Brunei Fertilizer Industries greenfield fertilizer complex will have a production capacity of 2200 tpd of ammonia and 3900 tpd of urea. After its planned completion in 2021, the plant will use parts of Brunei's large natural gas reserves as feedstock to produce high quality nitrogen fertilizer mainly for the export market.

thyssenkrupp's scope of supply for this fertilizer complex will include the engineering, supply of equipment, erection, supervision of construction and commissioning, as well as various offsite and related utility systems.

Brunei is one of the world's largest producers and exporters of natural gas. This project will support the government's long-term development strategy to diversify the country's economy. As one of the key investment sectors, the development of downstream activities in the petrochemical industry will help better utilise the main economic resources available: oil and gas.

CANADA K+S Potash Canada celebrates Port Moody terminal opening

K+S Potash Canada (KSPC) and Pacific Coast Terminals (PCT) have celebrated the opening of a state-of-the-art potash handling and storage facility at PCT's Port Moody terminal.

The port facility is the western port destination for potash from KSPC's new multi-billion dollar Bethune mine in southern Saskatchewan.

In 2014, KSPC and PCT signed a long-term agreement for the delivery of products from KSPC's Bethune mine to international clients. The agreement included modifications to PCT's existing facility, as well as the construction of a new potash storage building on the site. Now that these modifications have been completed, PCT's site is the most

innovative of its kind in the world.

Modifications to the site include a railcar unloading facility; underground and above ground conveyor belts; new transfer towers; and a 263 m storage warehouse with capacity for 160 000 t of product. The facility is able to unload a 18 000 t train in just four and a half hours. Ships with a capacity of 70 000 t can be loaded at the quay.

Potash from Bethune will be transported to the new facility in unit trains pulled by up to five Canadian Pacific locomotives, at a total length of approximately 3 km. Potash delivered to PCT's site will be destined for China, Southeast Asia, India, Brazil and other international locations.

ARGENTINA PhosAgro considers DAP sales to Argentina

Following the decision taken by Argentinian President Mauricio Macri to cancel the 6% import duty on high quality diammonium phosphate (DAP) from Russia, PhosAgro is considering increasing its sales of fertilizers to Argentina.

According to Andrey Guryev, CEO of PhosAgro, President of the Russian Association of Fertilizer Producers and Chairman of the Russian-Argentine Council of Entrepreneurs from the Russian side, this was a joint effort of federal authorities, including the Ministry of Foreign Affairs and the Ministry of Economic Development, as well as the intergovernmental Russian-Argentine commission for co-operation in trade, science and technology, the Russian Union of Industrialists and Entrepreneurs and the Chamber of Commerce and Industry. The interests of Russian business have been upheld by lifting restrictive measures on the trade of high quality Russian mineral fertilizer products.

"We are grateful to the Argentine authorities for the constructive dialogue and willingness to bolster mutually beneficial cooperation between Russian and Argentine

companies," Guryev commented.

Both Russian fertilizer producers and Argentine farmers will benefit from the development. Customers will benefit from additional volumes of 300 000 – 350 000 t of high quality DAP products that are free from potentially harmful contaminants.

Guryev explained that agricultural products and mineral fertilizers, which are necessary for increasing the efficiency of agricultural production, constitute one of the most important and largest areas of trade between Russia and Argentina. The Russian fertilizer industry views both Argentina and the rest of Latin America as a high-priority market, alongside the Russian domestic market. In 2016, Russian fertilizer exports to Argentina grew by 1.6 times year-on-year to 521 000 t, making up 43% of Russia's overall exports to Argentina.

"The illogical situation in which high quality Russian DAP, which is free from potentially harmful contaminants, was subject to import duties, while fertilizer containing harmful substances were allowed without being subject to duties, has now been resolved," he concludes.



WORLD NEWS

IN BRIEF

USA

AMECO has announced that it has finished the commissioning of a fit-for-purpose portal reclaimer for a major global producer.

The portal reclaimer can handle 480 tph of urea with a rail span of 54 m at a nitrogen operations facility in Texas, US.

AMECO designed and supplied an innovative urea storage solution with a portal reclaimer capable of dragging urea up to 20 degrees below ground.

The portal reclaimer reportedly allows the facility to increase its storage capacity by 50% compared to other solutions.

Canada

In order to match supply to market demand, and to utilise its lowest-cost Rocanville facility, Potash Corp. has announced that it will temporarily curtail potash production at two Canadian mines.

Allan will reportedly curtail production for a 10-week period, commencing 19 November 2017. Lanigan, meanwhile, will curtail production for an eight-week period, commencing 3 December 2017.

The amount of temporary layoffs associated with these adjustments has not been determined. Instead, the company says that it is assessing opportunities for reassigning employees during these periods to essential services, capital projects and maintenance activities.

India

The Arab Potash Co. has reached agreements to supply 550 000 – 600 000 t of potash to Indian Potash Ltd and Zuari Chemicals. These quantities will be shipped from Aqaba port to Indian ports in 2017 and in 1H18. The prices and terms of these contracts will be in line with market conditions globally and in India.

USA Vivakor acquires strategic intellectual properties from QSI

Vivakor Inc. has released a statement claiming that it has acquired strategic intellectual properties (IP) from QuantumSphere Inc. (QSI).

In an all-stock transaction, Vivakor secured patented Nanocatalyst IP from QSI. This technology produces a number of catalytic elements in the 30 – 60 nanometer range. Vivakor claims that the result is a catalyst that has a high surface area yielding greater catalytic activity. Approximately 90% of all chemical processes make use of catalysts and, due to the requirement for cleaner fuels and more efficient chemical production processes, worldwide demand for catalysts is forecasted at over US\$20 billion in 2019, according to Vivakor.

FeNIX is QSI's lead product, and is an iron-based nanocatalyst designed for use in ammonia production. It is applied as a coating to existing standard commercial ammonia catalysts and is considered

a 'drop-in' solution. This is key when introducing and commercialising a catalyst enhancement technology in the chemicals industry.

In 2015, FeNIX product efficacy was demonstrated in two commercial ammonia plants. The plants, located in central China, reported an improvement of up to 15%. Because of the increase in catalytic activity, higher production conversion rates can help to reduce consumption of energy and increase production output. As a result, this provides greater profits to chemical plant owners and operators.

In the statement, Vivakor claims that it will leverage QSI's patented capabilities and existing commercial relationships in order to help penetrate the US\$100 billion ammonia market. The company will also use QSI's IP to develop catalysts for other chemicals markets, including methanol, hydrogen, light olefins, and other hydrocarbon-based processes.

CANADA Two fertilizer terminals completed

Federated Co-operatives Ltd (FCL) has commissioned state-of-the-art, high-throughput fertilizer terminals in Brandon, Manitoba, and Hanley, Saskatchewan, Canada. Both facilities blend and distribute a complete suite of crop nutrition products for Co-op locations and agricultural producers throughout the Canadian Prairies.

The 68 000 ft² terminal in Manitoba has the capacity to house 27 500 t of fertilizer, while the second terminal in Saskatchewan is 96 000 ft² and has the capacity to store 45 000 t of fertilizer. The facilities were constructed on a combined budget of CAN\$75 million.

Construction of the facilities was announced in 2016 and was completed on schedule. Both terminals began receiving and shipping product in April. The Hanley facility currently has

five full-time employees, while the Brandon facility has four.

More than 125 Co-op Agro Centres across Western Canada currently provide agricultural producers with important crop supplies, including crop protection products and seed. Of those, 77 locations currently offer fertilizer and more are expected to include it among the products and services they provide in the future.

Each terminal can load a super B trailer of blended fertilizer in 10 min. and dispense up to 400 t of straight fertilizer in an hour. Rail access allows both facilities to efficiently receive product from domestic and international suppliers, while each terminal is centrally located for distribution to Co-op Agro Centres across Western Canada.

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WORLD NEWS

NEWS HIGHLIGHTS



- Ethiopia considers new partner in potash mine development
- Agua completes modelling and update of phosphate resource
- JBS invests in fertilizer plant in Brazil

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GERMANY Minister President of Hesse visits Werra Verbund plant

K+S has announced that Volker Bouffier, the Minister President of Hesse, Germany, has visited the Werra Verbund plant to gain a greater understanding of the current economic situation of the potash district.

During the visit, Bouffier also praised the company's efforts to make production more environmentally friendly using the latest technology. K+S claims that a focal point was the progress on developing a new kainite crystallisation and flotation facility (KCF) to decrease wastewater.

Bouffier said: "The state government of Hesse remains committed to creating a stable environment to safeguard jobs in the Werra potash district. However, we expect K+S to continue to stand by its obligation to deploy innovative processes and technologies to conserve the environment in the future as well."

Timo Lübeck, the local Bundestag candidate, noted that he was pleased that the minister president had accepted his invitation to visit the region, adding that it was "important to have Hesse's Minister President actually on the

ground and that potash miners could always rely on his word. A close dialogue with the Board of Executive Directors, plant management and employees is indispensable."

K+S claims that Bouffier was informed in detail on the status of work on the new KCF facility, which will reduce saline wastewater by a further 20% in future. In addition to this, he also asked about the extent to which the facilities erected as part of the initial package of water protection measures at the Werra plant sites had fulfilled expectations in practice.

The Chairman of the K+S Board of Executive Directors, Burkhard Lohr, emphasised that the Werra Verbund plant occupies a firm place in the global K+S Group production network. He said: "The extraction of domestic raw materials and the protection of the environment are compatible with each other. Our new facility will result in further innovations becoming reality. To make this perfectly clear, we are engaged in a close dialogue with political circles, our site local communities as well as environmental associations and civic initiative."

USA Mosaic provides detail on impact of Hurricane Irma

Mosaic Co. has released a statement providing an update on its third quarter business performance in connection with Hurricane Irma.

Before the hurricane, Mosaic claims that profitability metrics were originally tracking ahead of the third quarter guidance, with both good demand and strong operational performance.

Whilst the company says it was lucky not to have endured significant damages

from the hurricane, it does expect approximately 250 000 – 350 000 t of lower production in September. Added to the damage at the company's Bartow warehouse, Mosaic claims that this could result in a loss of up to 400 000 t of finished phosphate product.

The company will provide further details on the hurricane's financial impact in the month of September as part of its third quarter earnings release.

An Absolute Indication of HTHA in Parent Material...

The presence of Hydrogen in industrial plants is a source of damage. HTHA (*High Temperature Hydrogen Attack*) is a form of degradation due to the formation of Methane (CH₄) by reaction with carbon in steel. HTHA can occur either in the parent material or in the weld itself and manifests itself in several types of alloys.

Methane formed by the reaction accumulates at internal voids on the grain boundaries, where under certain conditions the build-up can produce micro-cracks. In the weld material this leads mostly to a leak before break situation but when the damage is in the parent material, the result is mostly catastrophic for people and the environment.

The inspection methodology uses advanced techniques including Time Of Flight Diffraction (TOFD), Advanced ultrasonic backscatter technique (AUBT), and velocity ratio measurements.

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For more information on HTHA detection please contact:

Dr Jonathan Burns | +44 (0)1954 231612 | jonathan.burns@mistrasgroup.co.uk

WORLD NEWS

DIARY DATES

Sulphur 2017

6 – 9 November 2017
Atlanta, USA

<https://events.crugroup.com/sulphur/home>

Energy Efficiency Conference 2017

13 – 15 November 2017
Athens, Greece

<https://ertc.wraconferences.com/>

Iranian Nitrogen + Syngas 2017

25 – 26 November 2017
Tehran, Iran

<https://events.crugroup.com/iraniannitrogensyngas/home>

Turbomachinery & Pump Symposia

12 – 14 December 2017
Houston, USA

<http://tps.tamu.edu/>

Nitrogen + Syngas 2018

26 February – 1 March 2018
Göteborg, Sweden

<https://events.crugroup.com/nitrogenandsyngas/home>

Phosphates 2018

12 – 14 March 2018
Marrakesh, Morocco

<https://events.crugroup.com/phosphates/home>

Middle East Sulphur 2018

18 – 21 March 2018
Abu Dhabi, UAE

<https://events.crugroup.com/middleeastsulphur/home>

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WORLD
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MAGAZINE

SAUDI ARABIA Production commences at Umm Wu'al phosphate project

According to Fluor Corp., Ma'aden Wa'ad Al-Shamal Phosphate Company's (MWSPC) Umm Wu'al phosphate project in Saudi Arabia has started production of ammonia, merchant-grade acid and fertilizer. MWSPC is a joint venture between The Saudi Arabian Mining Company (Ma'aden), The Mosaic Company and Saudi Arabia Basic Industries Corp. (SABIC).

Fluor is providing overall programme management services, as well as engineering, procurement and operations and readiness services for various scopes of this US\$8 billion megaproject.

"As part of Saudi Arabia's Vision 2030, this world-class project will have a long-lasting impact on the region, as it diversifies the country's economy and creates local job opportunities for citizens," said Tony Morgan, President of Fluor's Mining and Metals business. "After less than four years from the start of the execution phase, we are proud to have partnered with Ma'aden to

bring this facility to production. We look forward to continuing our partnership with Ma'aden in developing their next phase of mining projects in Saudi Arabia through our recently signed memorandum of understanding."

Production has begun on diammonium phosphate (DAP) fertilizer, merchant-grade acid and ammonia. Phosphate serves as a key element in fertilizer for agricultural crops. As one of the largest integrated phosphate fertilizer plants, the facility will help meet global food supply needs by delivering 3 million tpy of diammonium phosphate and nitrogen, phosphorous and potash fertilizers.

With a peak site workforce of 28 000 from more than 50 nationalities, Fluor implemented its world-class safety programmes, including its Life CriticalISM programme, to support the project. As a result of these programmes, the project has achieved more than 46 million consecutive work hours without a lost-time incident.

USA Montana to receive fertilizer plant by December 2017

Town & Country Supply Association has announced plans to open a 22 800 tpy fertilizer plant in eastern Montana, US, by December 2017.

The plant will be capable of blending 240 tpy, and will store approximately 9000 t of urea nitrogen as well as a 5000 t monoammonium phosphate (MAP) bin. Ammonium nitrate (AN) is not expected to

be used at the plant. A spur line will be installed for railcars (85 cars).

"We think we're going to be more timely in getting product out to be spread," commented General Manager Wes Burley, adding that the plant will "allow us to quickly produce quality custom blends based on what the soil tests recommended for various crops in huge quantities."

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PAYING THE PRICE

Neil Fleishman,

Green Markets, USA,

overviews the challenging global outlook for NPK prices.

Global commodity prices are currently in a super-cycle and, like most commodities, fertilizers (nitrogen, phosphorus and potassium; NPK) are also oversupplied. While global macro-economic issues are a factor, the dynamic that got the market to this point is straightforward: a sustained period of high fertilizer prices led to over investment. Long-term demand estimates at the start of the expansion cycle also failed to meet high expectations, compounding the current over capacity problem.

Questions arise regarding when this super-cycle will shift and how much price support would be necessary to do so. While each nutrient category has different market characteristics, the path out of oversupply is rooted in long-term demand growth for all three of the major commodities.

Potash

Potash is the most highly concentrated of the three fertilizer markets, with the bulk of world trade being made by only a few players operating out of Canada, Russia and Belarus, and exporting approximately 78% of globally traded product. For 2017, potash nameplate production capacity has been estimated to be approaching 87 million t versus an estimated mid-point demand of 62 million t. This nameplate capacity figure includes slack capacity in Canada, recent global capacity additions and any potential brownfield expansions. Against nameplate capacity, Canada currently controls approximately 37% of capacity, with Russia controlling 14% and finally Belarus with 12%.

It is important to note that whilst operational potash capacity is lower than nameplate capacity, there is still significant oversupply. While it would take lead time for a company running at reduced rates to ramp back up, significantly higher prices and/or increased demand would pull product into the market, given enough lead time.

The Russian and Belarusian Trade Association (BPC) dismantled nearly four years ago and doing so decreased prices sharply in a move towards



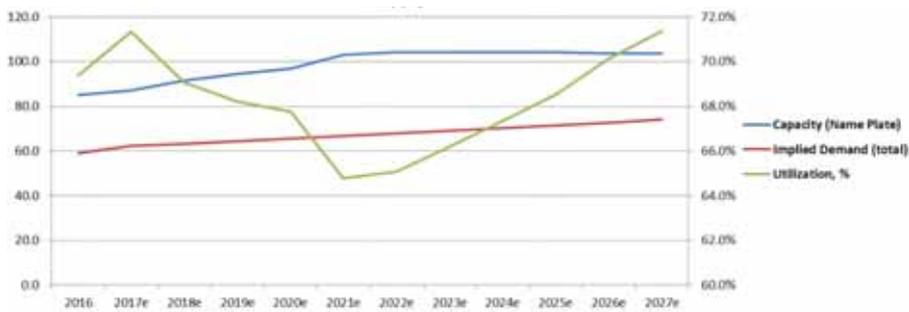


Figure 1. Global potash supply and demand.

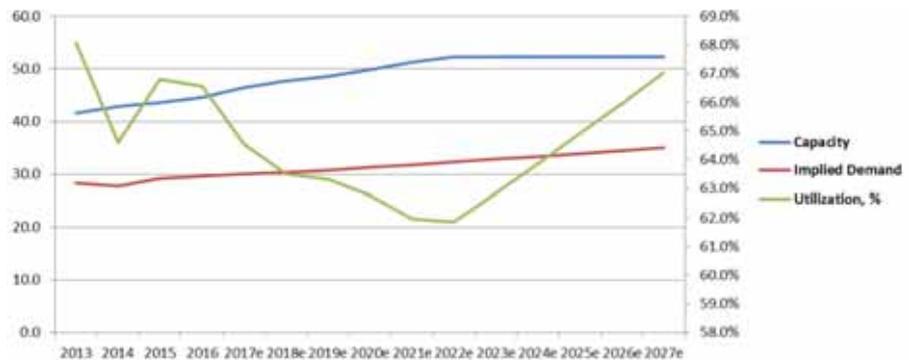


Figure 2. Global DAP/MAP supply and demand.

volume-over-price marketing. Before BPC dissolved, the two major export associations (BPC and Canpotex, the Canadian export arm) focused on price-over-volume marketing. Canpotex members currently take a disciplined approach to their production, whereas former BPC members run their production levels at maximum levels in a bid to pick up market share.

Many players in the market continue to watch for signs of improved co-operation between Russia and Belarus, as this would increase prices. However, this revived collaboration is likely elusive for the foreseeable future given long standing differences between the two countries. Belarus' Alexander Lukashenko has noted that the country's interests must be forefront in any potential agreement. For the moment, it remains clear that both sides are far apart on the negotiating table for near-term co-operation. A return to price-over-volume marketing strategies for all major players in the market is, therefore, unlikely.

Low prices have also led producers to capacity rationalisation and consolidation, with companies such as PotashCorp and Intrepid shutting in their higher cost capacity. The PotashCorp and Agrium (Nutrien) merger is a prime example of companies joining forces in order to capitalise and shield against unfavourable market conditions.

The global potash market's long-term pricing outlook suffers from significant slack capacity from established players combined with a capacity overhang. Recent projects that are now online include K+S' Bethune (formerly Legacy) mine in Canada at 2 million t and Tajikistan's Garlyk deposit at 1.4 million t. There is also incremental capacity on the horizon from multiple greenfield projects, for example from Eurochem, and over the next decade from Belaruskali, Uralkali and BHP Billiton – all of whom have substantial projects either under evaluation or progressing. China's

largest producers also continue to expand production. Moreover, there is also no shortage of junior players looking for funding as over 55 such projects have been tracked as of mid-July (not including dormant efforts). While supply management could potentially push potash prices higher, it would ultimately incentivise even more capacity, slack or otherwise, to enter the market.

For 2017, Green Markets continues to track solid potash demand globally. However, historically, this potash demand is highly elastic and suggestive of demand being predicated on a low-pricing environment. With the potash market now facing a long-term threat of new capacity, it will be a tough road to sustained higher pricing.

Phosphate

Phosphate supply has medium concentration but large players (Mosaic, Ma'aden and OCP) control over 25% of the global diammonium phosphate/

monoammonium phosphate (DAP/MAP) capacity. Green Markets has estimated that DAP/MAP nameplate capacity for 2017 will reach approximately 46.5 million t (nutrient) versus mid-point demand of 30 million t (nutrient). North America currently accounts for approximately 15% global capacity, with Africa at approximately 14% and China at approximately 31%.

Over the past decade, Ma'aden and OCP built mega-projects that brought close to 8 million t into the market. OCP, located in Morocco, is now the world's largest producer of both processed phosphates and phosphate rock. Notably, both OCP and Ma'aden plan continued expansion, predicted to potentially add another 7 million metric t of additional capacity in the next five to seven years (depending on financing). At current demand levels, this incremental supply (along with other potential projects) would continue to push the usage rates lower in the near to medium-term. Supply management from some of these players has been effective in creating price support in recent years and these expansions are likely to be managed to continue to reflect this discipline.

Historically, North America was one of the key producing phosphate regions. North American production has been declining in recent years due the region's high relative cost structure compared to the Middle East and North Africa. North American producers have shifted some production towards higher value specialty products, a trend that is unlikely to reverse. China remains one of the world's largest producers and exporters. In 2016, Green Markets tracked record exports from the country. With capacity still projected to grow further in China, excess exports from the country could continue to weigh on global pricing.

The current pricing environment has also led to consolidation in the phosphate market with Mosaic buying Vale's assets. Further consolidation in the market could be

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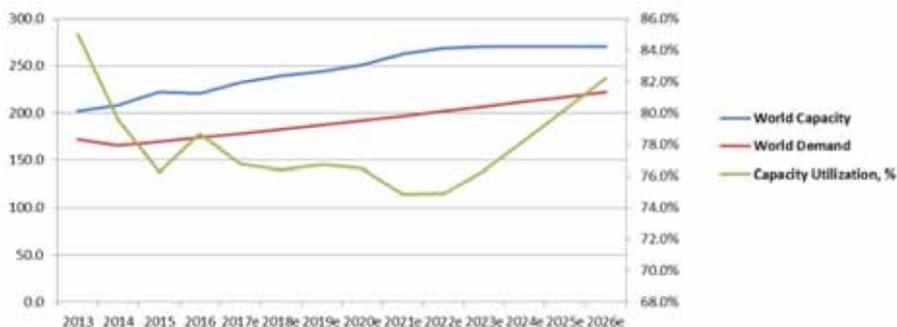


Figure 3. Global urea supply and demand.

on the horizon but vertical integration, synergies and a low cost of production would be key.

It must be noted that multiple DAP/MAP projects (outside of China) have faced significant issues, leading to only a handful with a clear path to increased supply. There are multiple reasons for this: a low price environment with current oversupply; announcements by the largest players of their goals for continued expansion; and political difficulties in the regions of intended projects.

Overall, this suggests that the phosphate market will begin to bottom heading into 2020. Green Markets forecasts that it will take several years for rates to recover meaningfully and provide price support.

Urea

Urea is the least concentrated of the three major nutrient markets with some of the world's largest producers only accounting for fractions of global supply. Supply management from producers would have almost no impact on global pricing. Global urea capacity is estimated to reach approximately 232 million t (including China) in 2017 versus mid-point of demand at 180 million t. Consolidation in nitrogen markets has been limited following fragmentation in the market and has diminished the impact mergers would have had on supply volumes.

Urea is the most ubiquitous nitrogen fertilizer globally, largely due to its ease of application and relatively low cost of transportation. The vast majority of the world's recent nitrogen expansions has been focused on urea in low cost areas of production. China is the world's largest global urea producer and is also thought of as the marginal cost supplier to the world.

China has been through a period of rapid capacity expansion over the past 15 years with capacity doubling. The country now accounts for approximately 40% of global urea capacity. With low prevailing global urea prices, China has moved to curtail production and permanently shut down less efficient capacity. Nearly 70% of the nation's urea capacity is based on coal gasification, which has led to environmental concerns. As steps are taken to limit production and curtail exports, the importance of Chinese product on the global market has significantly reduced with global expansion picking up the balance.

North America has been a centrepiece of the global nitrogen expansion: with a sustained outlook for low-priced natural gas feedstock, there have been over 40 potential projects announced in the past five years. While many have

long since been shelved or cancelled, the first wave of new production capacity has already impacted markets and led to a reduction in imported product.

This increased localisation of production in North America is a global trend. Multiple countries, including India, are also looking to decrease dependency on imported product. However, feedstock sources remain a challenge. Notably, export availability from other regions (outside

of China), such as the Middle East, North Africa and Southeast Asia, have also increased, leading to a glut of urea on the global market at the same time that import needs are being reduced.

This expansion in urea capacity pushed usage rates lower and reduced the need for Chinese exports. During 2016, the market saw a reduction in exported Chinese product coupled with even lower prices. This trend has continued into 2017. While the pace of expansion in urea capacity is beginning to slow, Green Markets is tracking a backlog of projects that could continue to take shape if prices were to recover. Any meaningful near-term recovery in urea pricing would also likely pull Chinese product into the market, muting any upside to pricing.

Most importantly, the market is faced with the reality of the supply and demand balance for urea. In 2013, the global utilisation rate of urea production was 85%; in 2017, Green Markets estimates it has fallen to 76%. Meanwhile, benchmark prices in the US have fallen by as much as 32% in 2017, and it is thought that the supply and demand balance will bottom into 2020, moving slowly and only slightly higher from there. Thus, while the pace of supply expansions is curtailing, the market will be challenged to absorb the significant supply increases of the last few years.

Conclusion

The current low price environment for primary fertilizers is likely to continue in the near-term and the market is likely to stay challenged as a result of significant over capacity. Fertilizer commodities are in a super-cycle of oversupply and the path out is long-term global demand growth alongside a slowdown in capacity expansion. While each nutrient has its own dynamic with regards to market concentration and potential for supply management, all three are facing oversupply with potential continued expansion on the horizon.

The potash market has the highest concentration of players, fractured supply discipline, and faces a capacity overhang for the foreseeable future. The phosphate market has medium concentration of players with several large established companies who continue to build supply. The nitrogen (urea) market is highly fragmented and significant capacity growth means it will take time for demand to catch up. Many urea global urea projects that are currently stalled or in difficult regions to build could come to fruition if prices were to rise. Nevertheless, recovery may take longer than some in the market are professing and the path higher will likely be muted when that happens. **WF**

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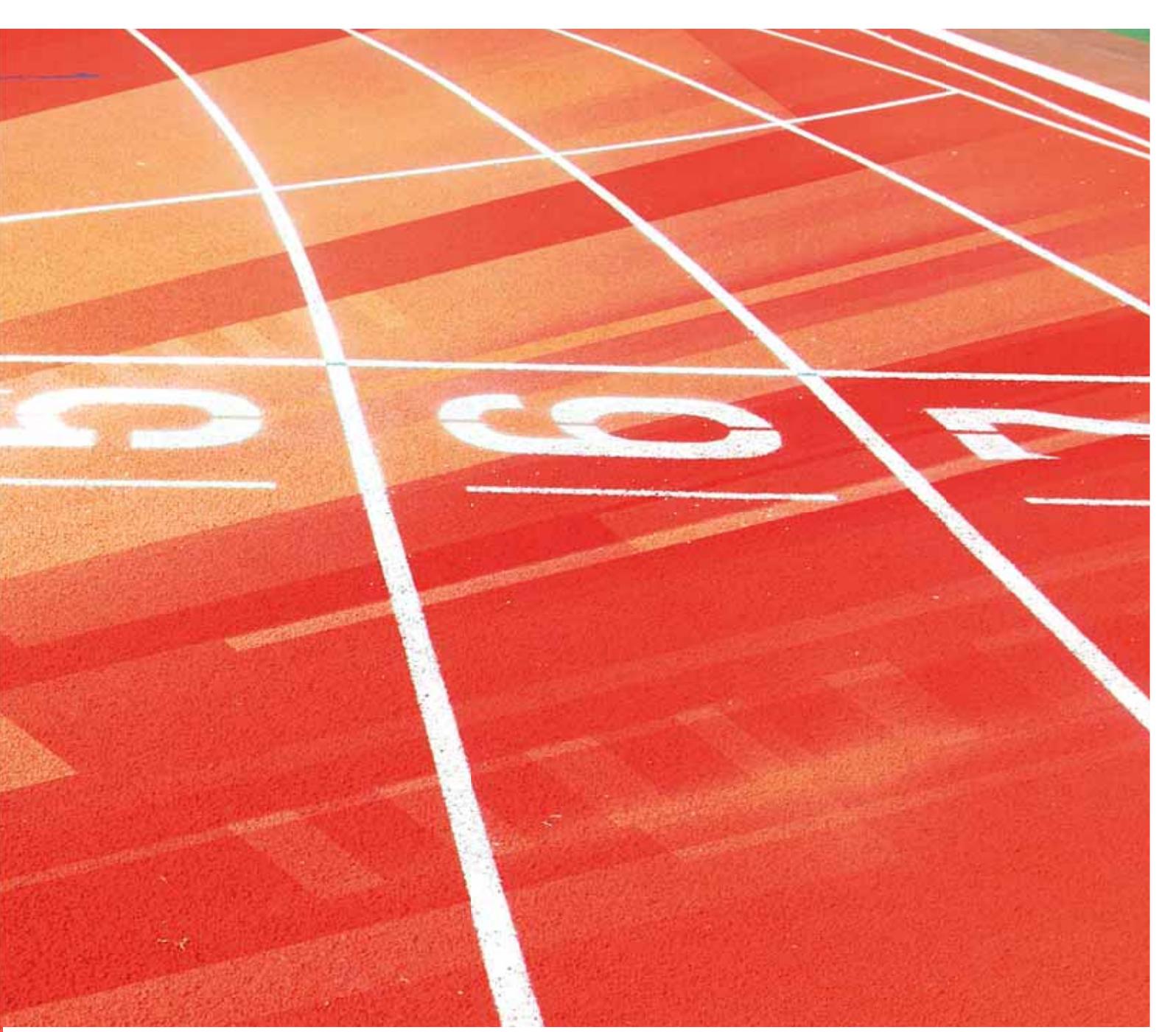
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KEEPING PACE



Meena Chauhan, Integer Research, UK,
provides an outlook on the global sulfur and sulfuric acid market.

The global sulfur market has seen significant growth in recent years, on the back of the rise in demand for energy and petrochemicals. There has been a marked drive in investment in the sour gas and heavy oil sectors, which has led to increased sulfur recovery across the world. Due to the hydrogen sulfide (H_2S) content in sour gas, the scale of growth in gas processing has been a leading factor in supply changes in many regions. A main shift in the sulfur industry over the last decade has been a rise of sulfur production in the Middle East region. This has led to changing dynamics across global pricing benchmarks and trade flows.

In 2016, Middle East sulfur production totalled over 16 million t, representing approximately 27% of total

global supply and a 66% increase on regional supply since 2011. The region overtook North America as the leading sulfur producer in the world last year, with a large scale sour gas project in the United Arab Emirates (UAE) adding an additional 3 million tpy of sulfur capacity alone. The UAE has become the largest sulfur producing country in the Middle East, with its supply exceeding 6 million tpy. Due to limited domestic consumption of sulfur in the UAE, trade patterns have shifted. Increased volumes are now being exported to markets such as Brazil, China and North Africa.

Medium and long-term sulfur outlooks

The medium-term outlook for global sulfur supply is for strong growth, with projects that are already in the

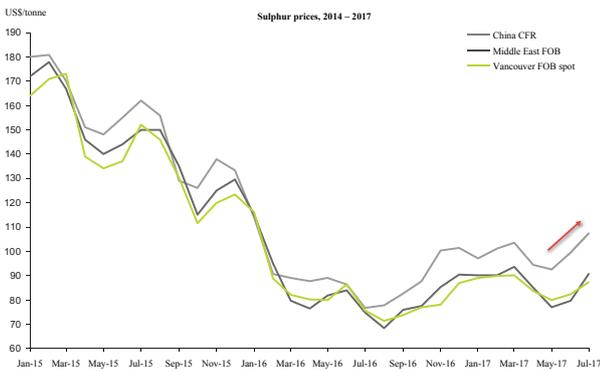


Figure 1. Global sulfur prices have been firming since May as demand in key markets including China emerged.

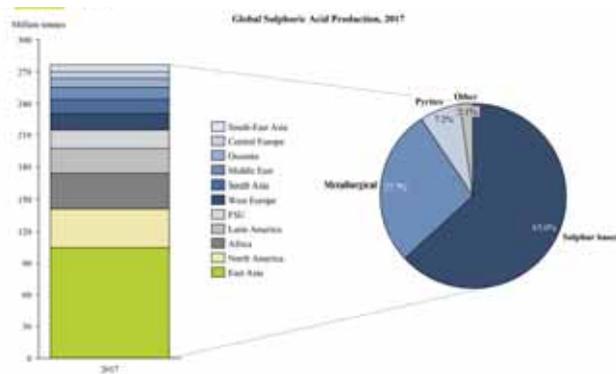


Figure 2. Sulfur-based acid production gained market share in 2017 to represent 63% of global output.

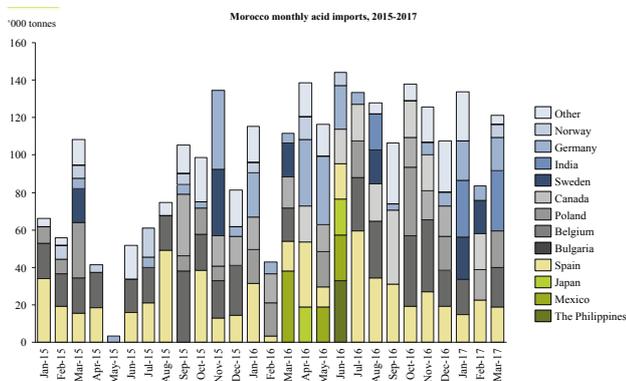


Figure 3. Moroccan sulfuric acid imports have remained strong in 2017, with 1Q17 up by over 25% year-on-year, continuing to support European acid prices.

pipeline due to come online in the period up until 2022. In Kazakhstan, the long-delayed Kashagan project has started oil production. This is set to add over 1 million tpy of sulfur at capacity. The first sulfur export shipment is expected in late 2017. If the project ramps up to capacity on schedule, this points to a more bearish sentiment for the market due to increased availability of supply.

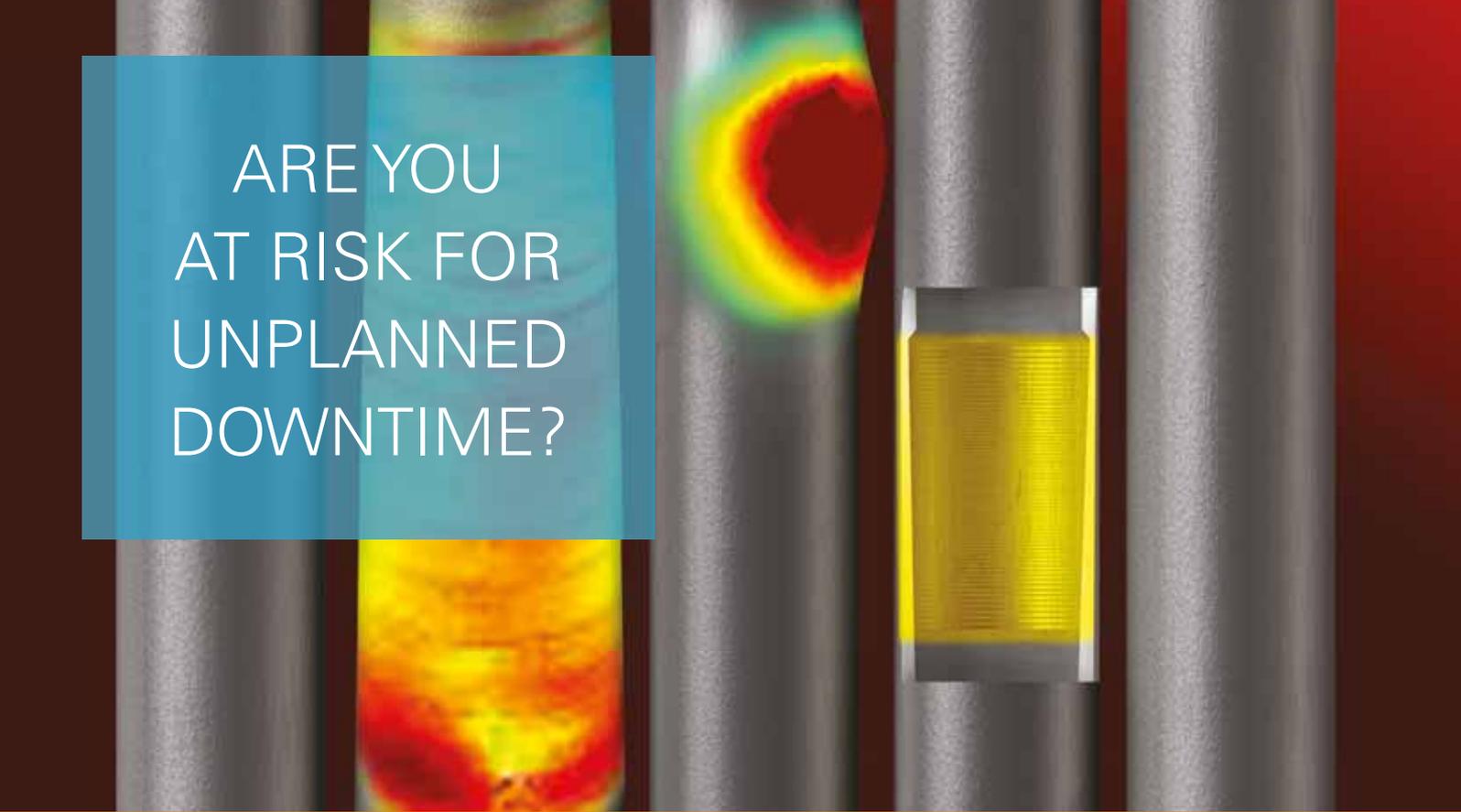
Another project on the horizon that is set to add to the global supply balance significantly is RasGas' Barzan project in Qatar. There is uncertainty around the

potential ramp-up date of this project, with numerous technical delays pushing the startup timeline forward. Integer now expects the first phase of the project to begin production in 2018. The project comprises two phases, due to start in quick succession of one another. At capacity, Barzan would add over 800 000 tpy of sulfur, impacting the export market significantly. Based on expected project developments in Qatar, Iran, the UAE and Saudi Arabia, the Middle East is set to remain the leading global producing region of sulfur in the medium to long-term. As a result, increased competition is likely to emerge between suppliers across all regions due to the lower cost of moving sulfur from plants to ports for most Middle East producers, versus higher logistical costs for some producers in other regions such as the Former Soviet Union and North America.

One issue impacting long-term developments for sulfur supply is the sustained low oil price environment. This has taken its toll on some refinery and energy projects, with many cancellations or delays due to investment freezes. Oilsands development in Western Canada has seen a drop, as well as some refining projects in Asia. This slowdown in investment signals the potential for lower growth rates for sulfur supply in the long-term and a tighter balance in the market. However, in the medium-term, almost every region is set to see supply increases.

Western Europe and North America are the exceptions to this trend. While the US is set to remain the leading global producer of oil-based sulfur supply over the next 10 years, gas-based supply is expected to see a dip in the country. This trend is mirrored in Canada, where there has been a structural decline in this sector for many years, leading to a net decline in total sulfur production. Sour gas well depletion and challenging economics have been drivers of this downturn. Meanwhile, in Western Europe, gas-based production is also expected to decline, and there is little scope for any growth from the oil refining sector. In recent years, numerous oil refineries in the region have closed or been converted to terminals in the region, creating pockets of tightness.

The majority of elemental sulfur is used to produce sulfuric acid in the production of processed phosphates. Sulfuric acid is also used in the chemical industries, including caprolactam and titanium dioxide, with numerous applications outside the fertilizer sector. The main hotspot for sulfur demand growth is the Middle East, North Africa (MENA) region due to the expansion of processed phosphates production. In Morocco, OCP's Jorf Lasfar hub expansions have led to changes in sulfur and sulfuric acid trade to the country due to the absence of domestic sulfur production. In Saudi Arabia, processed phosphates production is also set to rise significantly, leading to a 100% increase in domestic sulfur consumption in the medium-term. The country is also the leading sulfur supplier to China, reaching 2.5 million t in 2016 on this trade route. The changing tide of demand in Saudi Arabia has led to questions over whether it will maintain its ranking in China's trade. Based on production expectations from



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planned projects, sulfur output would exceed 6 million t in the outlook and offshore exports would likely remain strong.

Global sulfur prices have been mixed through 2017. A weak 2Q17 weighed on prices, which gave way to a firmer start to 3Q17 due to improved import demand in China. Low refinery run rates led to an erosion of inventories, and buyers re-entered the market. The run up in pricing is expected to continue in the short-term. However, going into 2018, there is a question over how much additional supply will enter the market to influence the global balance, which may put downwards pressure on pricing. Project delays could keep prices in a higher range.

Sulfuric acid

Global sulfuric acid production totalled close to 268 million t in 2016, with over 60% produced from elemental sulfur. This share of supply largely represents consumers producing acid for captive consumption across the fertilizer and industrial sectors. In some cases, sulfur-based acid is produced solely for sale and trade. Approximately 28% of global acid supply is metallurgical based, produced as a byproduct from the metals sector. It is this portion of the market that has the most impact on global trade and sulfuric acid pricing. Despite the scale of acid produced worldwide, only around 8 – 9% of total output is actually traded. Other than metallurgical and sulfur-based acid, the remaining portion of supply is split across other types, including acid from pyrite ore roasting – this is the one source of acid expected to contract. In 2017, Integer estimates pyrites represented 7% of global supply, which is set to decline to 5% in the medium to long-term. This is driven largely by changes expected to materialise in China due to the rise of other forms of domestic acid production.

China is currently the leading global producer of sulfuric acid, with its share of metallurgical supply also set to grow significantly due to the spate of investments in copper smelters. While China is a net importer of acid due to the size of its consumption needs, volumes of acid are also exported during periods of high demand or high pricing. China's acid supply is forecast to rise by 2% in the medium-term, by approximately 1.8 million t in volume terms. Acid imports to China average around 1 million tpy. A key question for the acid industry going forward is whether demand in the country will also grow apace with new supply. Along with the expected slowdown in new capacity additions in China's fertilizer sector, overall acid consumption growth rates are expected to slow in the medium-term view. Any surplus acid in the market would thus mean increased potential for a rise in exports.

Global sulfuric acid prices have been firming through much of 2017, with major benchmarks edging up on the back of stretched supply. European export prices crept up into the high-US\$20s/t FOB in 3Q17 as deals into Latin America boosted the market. Major smelter turnarounds have been limited in Europe in 2017, but prices have been buoyed by changes in availability, a reflection of tight supply and healthy demand. Supply issues in Latin America have had a major impact on global benchmarks due to a spate of spot purchases from Chile.

Chile's acid imports have been declining in recent years, owing to shifting demand from the copper sector. Since 2013, annual trade to the country has dropped from over 3 million tpy, to just 1.7 million t in 2016. However, in 2017, this trend has seen a complete reversal due to major outages from smelters in the region, including Peru. As a result, acid imports to Chile saw an 18% increase year-on-year in 1Q17. January – May 2017 data shows a 6% rise, as the situation began to normalise. In the coming years, Integer expects that sulfuric acid consumption in Chile will drop, leading to further decreases in acid imports.

For the remainder of the year, support continues for sulfuric acid prices to remain stable to firm, driven by tightness in major regions. Northeast Asia is set to remain short of spot availability throughout 4Q17, keeping Japan and South Korea's sulfuric acid export prices firm. Trade data for the year to date reflects how both planned and unplanned outages at smelters have impacted availability and prices so far. Trade from Japan in January – May 2017 totalled 1.1 million t, representing a 10% drop on a year earlier, adding to the tighter balance in the region. Meanwhile, South Korean acid exports have also dipped, with 1H17 data showing a 7% drop to 1.4 million t.

Sulfuric acid trade to Morocco has seen a major shift in recent years, and this is set to remain an influential factor for the market in the short to medium-term. Historically, direct acid imports to Morocco were in the region of 400 000 – 500 000 tpy. Since 2014, this has been on the rise and more than doubled in 2016, with trade totalling 1.3 million t. Supply sources have also been evolving along with the volume increase, with 2016 trade, including Canada, the Philippines, Japan and Mexico. European sulfuric acid producers are reliant on this market as an outlet for surplus acid, particularly with the loss of Cuba, a major market. The Moa nickel and cobalt project in Cuba previously imported part of its sulfuric acid requirement, with annual imports totalling between 400 000 t and 500 000 t. However, since the startup of a new 2000 tpy sulfuric acid plant at the project in 2016, imports have dwindled, leading to a displacement of this volume from countries including Spain, Germany and Italy.

Conclusion

Regarding sulfuric acid demand, Integer expects to see growth in almost all regions for the fertilizer sector in the medium-term, with the exception of North America and Central Europe. It also assumes that there are limited prospects for demand growth in China as capacity at processed phosphates fertilizer plants continue to operate at low levels due to ample local supply. The main hubs for sulfuric acid consumption growth will be Africa and the Middle East. However, the trend in the Middle East would not impact acid trade due to its captive production of sulfuric acid from sulfur. The non-fertilizer sector meanwhile is estimated to grow by 8% in the medium-term. East Asia, led by China, is set to see growth in this area in the forecast, representing the largest share of regional consumption at approximately 30%. **WF**

A FOCUS ON EFFICIENCY

Catalyst gauze systems for ammonia oxidation and N_2O reduction are a key component in nitric acid plants.

Oliver Henkes, Heraeus Precious Metals, Germany, illustrates this via a series of technical case studies.

To ensure that a catalyst gauze system provides high performance for ammonia oxidation, nitrous oxide (N_2O) reduction is an important factor for nitric acid plants. When used in nitric acid reactors, the structure of the catalysts changes, improving their efficiency. Over a period of several months, however, catalytic activity diminishes as the surface of the gauzes becomes contaminated and their structure modifies. Because of this, the gauzes must be replaced periodically. This is the normal way of life for any platinum gauze. Besides that, high quality technical service from the gauze supplier can make a huge difference for nitric acid plant efficiency.

Plant improvement projects or limiting production losses after an unexpected incident are examples of beneficial technical service. Particularly in emergencies, quick and reliable feedback from the supplier is of the utmost importance in order to detect and eliminate the root cause of the problem and quickly bring the plant back to normal performance levels. Technical service teams for customers in America, Europe, Africa, and Asia/Australia are available to customise this gauze and catalyst system solution, install it on site and check the process parameters while in operation.

This article provides a series of case studies, which demonstrate the technical services that Heraeus has provided in recent years. The examples cover various scenarios, from mechanical damages or contamination of catalyst gauze systems, to partial replacement of gauze packs with inadequate laughing gas emissions or the development of scenarios for possible root causes of unusual low gauze efficiency and high N_2O emissions. The article will investigate the process data and environmental conditions followed by working out action plans depending on the situation, which only becomes visible after plant shutdown.

A further area where supplier support is a key factor is in secondary catalyst N_2O reduction projects, including planning, designing, manufacturing and installing catalyst baskets. Secondary catalysts have been installed by Heraeus in numerous reactors, with satisfied customers running the company's secondary catalyst technology for over 10 years to reduce up to 95% of N_2O . N_2O is generated as a byproduct during the ammonia oxidation. More than 60 million t of nitric acid are





Figure 1. Repairing a torn gauze pack.



Figure 2. The efficiency drop within the campaign.

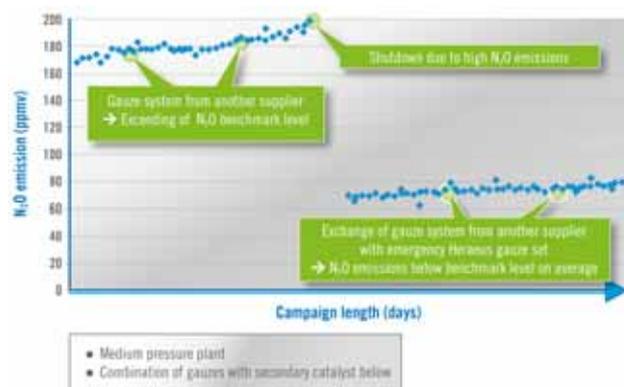


Figure 3. N₂O emission increase after changing the gauze system.

produced across the world annually, with an estimated 500 000 t of N₂O – the equivalent to carbon dioxide emissions from over 60 million mid-sized cars.

Performance loss in a plant

Repairing a torn gauze pack

In one case, a customer reported an incident that happened during the ignition procedure of its plant. The gauze pack was damaged by the rotating gauze ignition system due to them being in close proximity. The gauze showed a large tear over almost the entire diameter, with a few smaller tears in other areas of the gauze. As the customer did not hold any spare gauze material and because of bureaucratic obstacles for short-term precious metal imports into the country, an immediate supply of gauze material on site for repair or exchange was not possible.

The only remaining option was to repair the gauze without any additional gauze material. To enable a quick restart of nitric acid production, a Heraeus engineer was sent to the customer with welding equipment. After a day of precision work, the torn gauze pack was fully repaired, and slightly stretched again to the

necessary diameter, enabling an immediate restart of the plant. This emergency solution, in turn, allowed for continuous production at a reasonable performance until the new gauze system was supplied to the plant.

Studying the root causes of an efficiency drop

After about one-third of the nominal campaign, a customer reported that the conversion efficiency showed a sudden decreasing trend, from more than 96.5% to below 94% within a few days. An emergency shutdown took place in this phase, but it did not seem to be related to the efficiency issue. Days later, the situation slightly improved back to nearly 96% before it worsened again to almost 93%. The N₂O emissions showed a corresponding behaviour with increasing N₂O at lower conversion efficiency.

Generally, the following reasons could have been responsible for the observed behaviour and were therefore discussed:

- Contamination of gauzes from the process gas.
- Lower gauze temperature.
- Mechanical damage of the gauze system.

According to the customer, the process parameters and plant load were similar to the previous campaign where this behaviour was not noted. The campaign did not show an unusual amount of stops and the gauze pack had no damages through the inspection glass.

The focus was then put on the environmental conditions outside of the plant during the timeframe of the performance disturbances. It was reported that, along with the efficiency changes, a sandstorm was noted in the plant area. Because the filter exceeded the maximum specified operation pressure drop, some air filter cartridges underwent an online replacement once the storm had calmed down. This made potential contamination of the gauze pack more likely and, as the efficiency reduced further to 87%, it was decided to open the burner hood. Although a contamination now seemed to be the clear reason for the issue, it was still not clear which kind of contamination had harmed the gauze pack. Therefore, two scenarios and actions were defined:

- Contamination with loose sand particles: in this case, a procedure for careful vacuum cleaning without directly touching the gauze pack was given.
- No visual contamination of the gauze pack: in this case, micro-scale contaminants were expected to be responsible. For this, an instruction for pickling (treatment of the gauzes with acid solution) of the gauzes was provided.

Upon opening the burner, it was immediately clear that high levels of sand were evident on the gauze pack, which resulted in the performance drop. The dust was, therefore, carefully removed by vacuum cleaning. After the catalyst pack was reinstalled, the plant was started up again and went back into operation with the previously recorded conversion efficiency of more than 96.5%.

N₂O abatement projects: secondary catalyst basket implementation

In 2012 – 2013, Heraeus installed several catalyst baskets for nitric acid plants. These plants either had no secondary catalyst installed beforehand or the installed solutions until then were not suitable to reach a sufficiently low N₂O emission level that would either

fulfil certain N₂O emission limits or generate a sufficient amount of carbon credits.

Heraeus offers the following N₂O abatement services within ammonia burners:

- Designing secondary catalyst and containment systems to fulfil customers' N₂O targets.
- Inspecting the current situation in the burner and identifying principle solutions, depending on space restrictions or pressure drops.
- Putting together basic and detailed design proposals with its engineering partners.
- Selecting suitable manufacturing partners and inspecting manufacturing processes.
- Supervising of basket installation.
- Installing secondary catalyst and catalyst gauze systems.

These services allow customers to have a single partner that is responsible for all components of N₂O abatement (gauze system, secondary catalyst and catalyst basket). This assures the customer that no time is wasted on the identification of responsibilities between different parties. Instead, immediate solutions are developed to re-establish a high-level performance in case of problems.

Increased N₂O emissions

A medium-pressure nitric acid plant in Europe was originally equipped with a Heraeus FTC gauze system for low N₂O emissions and a below installed secondary catalyst. The nitric acid producer decided to change the catalyst gauze technology, deviating from the FTC gauze system.

With this system and the secondary catalyst underneath, typical N₂O emissions were 50 – 60 ppm by volume at campaign start and approximately 150 ppm by volume at campaign end. With the new gauze system, the initial N₂O emissions were already at a level of 170 ppm by volume at campaign start, which would have meant exceeding the EU-ETS III benchmark level from the beginning.

An emergency Heraeus gauze set for a partial system exchange was then designed and supplied within one week. With the help of the partial gauze system exchange, N₂O levels were brought back to levels that enabled the plant to stay below the N₂O benchmark level on average.

Conclusion

Normally, any platinum gauze for nitric acid production must be replaced periodically after several months. This is because catalytic activity diminishes the surface of the gauze. However, problems with the effectiveness of the gauzes can also occur during operation, such as mechanical damages or contamination of catalyst gauze systems, partial replacement of gauze packs with inadequate N₂O emissions or the development of scenarios for possible root causes of unusual low gauze efficiency and high N₂O emissions.

In all of these cases, high-quality technical service from gauze suppliers, such as Heraeus, can make a huge difference to nitric acid plant performance. This is particularly the case in emergencies where quick and reliable feedback from the supplier is of the utmost importance to detect and eliminate the root cause of the problem and quickly bring the plant back to normal performance levels. **WF**



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MAKING A DIFFERENCE



Mårten Granroth, Haldor Topsoe, Denmark,
explains how using the right catalyst can affect
sulfuric acid production.



Historically, many different methods for producing sulfuric acid have been used, perhaps most notably the lead chamber process. However, since the early 20th Century, the vast majority of sulfuric acid has been produced using different varieties of the so-called ‘contact’ process. The contact process is ‘so-called’ because the central concept is to react sulfur dioxide (SO₂) to sulfur trioxide (SO₃), a precursor to sulfuric acid, in contact with solid catalyst pellets. While platinum-based catalysts were originally used, their associated high cost and catalyst deactivation issues meant that they were replaced by vanadium pentoxide catalyst. Due to its comparatively low price and robustness, vanadium pentoxide-based catalysts have continued to be the industry standard since they were developed in the early 20th Century.

Most contact sulfuric acid plants today are dry gas plants, meaning that they operate with gases free of water vapour. This is achieved by drying the air that is used for sulfur combustion, or, in metallurgical plants, by drying the SO₂ gas before processing it. Sulfuric acid is produced in such dry gas plants by



Figure 1. A wet gas sulfuric acid (WSA) plant designed by Haldor Topsoe.



Figure 2. Different sizes and shapes of sulfuric acid catalyst.

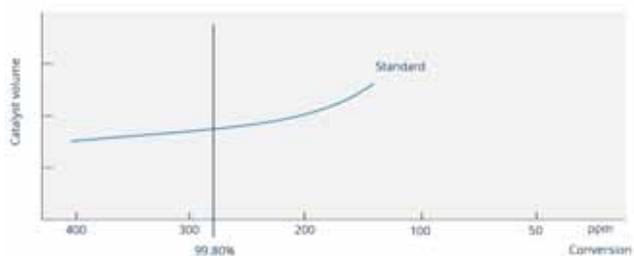


Figure 3. The increased catalyst demand for a sulfur burning plant that is running 11.5% SO_2 .

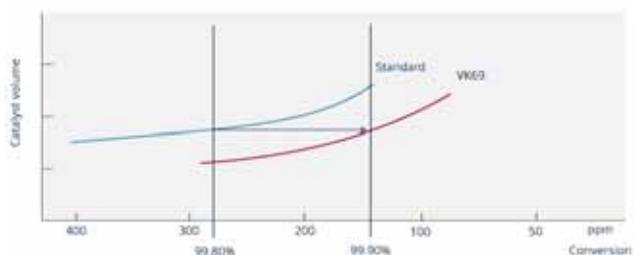


Figure 4. The difference in achievable emissions at different loadings between a standard catalyst and VK69 loadings for a sulfur burning plant running 11.5% SO_2 .

absorption of SO_3 in sulfuric acid, but this absorption would not work properly with water vapour in the gas.

Since the mid 20th Century, some contact sulfuric acid plants have been designed as wet gas plants. In these plants, water vapour is not removed, but instead passes through the plant. In such plants, sulfuric acid is produced by gas phase reaction between SO_3 and water vapour. The sulfuric acid vapour and subsequent condensation of the sulfuric acid

vapour forms liquid sulfuric acid. Wet gas plants, such as those based on Haldor Topsoe's wet gas sulfuric acid (WSA) process, are mainly used for smaller capacity plants. They are also used in special applications, such as hydrogen sulfide (H_2S) gas treatment and spent acid regeneration, where the removal of the water vapour would result in dilute sulfuric acid production, bad operation economy or production of unproportionally large amounts of waste water. Figure 1 shows a WSA plant.

Regardless of whether a plant is based on dry or wet gas, vanadium pentoxide catalyst is used today to convert the SO_2 to SO_3 . Having been around for more than a century does not mean that the vanadium pentoxide catalysts have not improved. Vanadium pentoxide catalysts for sulfuric acid production are available in a number of the different shapes, sizes and formulations to maximise the performance of acid plants. Figure 2 provides an overview of different shapes and types available for Haldor Topsoe's VK sulfuric acid catalyst series. Being at the heart of sulfuric acid plants, the choice and optimisation of catalyst loading will influence everything from cycle length, power consumption, steam production, production capacity and stack SO_2 emissions.

Minimising stack emission

High conversion levels of SO_2 to SO_3 have always been important for sulfuric acid plants that are based on burning sulfur to maximise sulfur utilisation and reduce costs. However, this is only true to a certain degree since the increasing costs that are associated with achieving higher conversion will eventually outweigh the sulfur cost savings. With an awareness of the environmental issues relating to acid rain in the 1960s and 1970s, there has been an understanding that SO_2 conversion needs to be higher than what is justified by plant economics.

Sulfuric acid producers across the world are facing more and more challenging emissions legislations. Plants that were originally designed for approximately 99.7% conversion are now asked to meet conversion levels of 99.9% or more. Therefore, something in these plants needs to change to allow them to continue operating. Simultaneously, with increasing legislative demand on emissions, competition within both the general bulk chemical market and the sulfuric acid business in particular is fierce. Plants need to run as efficiently as possible in order to stay profitable. However, the two are often not compatible, as reducing the cost per tonne of acid produced or gas volume treated means trying to maximise productivity for the plant. Emissions reduction, on the other hand, require production capacity cuts, expensive revamps or operating scrubbers, which add to operational costs and potentially add waste. A solution that is considerably less expensive than other options is to upgrade the catalyst loading in the plant.

Increasing catalyst volumes

A basic solution for improving catalyst performance is to simply increase the volume. If there is room in the converter and there are no temperature limitations that force the use of a more advanced catalyst, it will be both an effective and relatively inexpensive way of boosting SO_2 conversion. As more and more catalyst is added, higher conversion gets exponentially harder, as Figure 3 shows in a sulfur burning plant.

The main issue for most older plants is that their capacity has been increased multiple times. The effect of this is that



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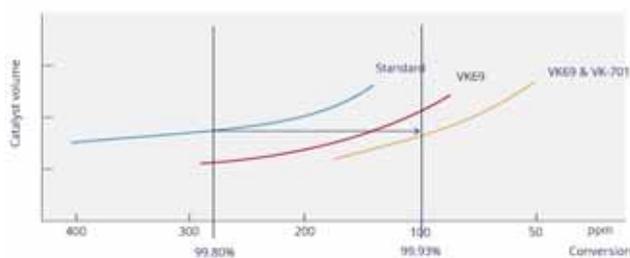


Figure 5. The difference in achievable emissions at different loadings between standard catalyst, VK69 and LEAP5 loadings for a sulfur burning plant running 11.5% SO₂.

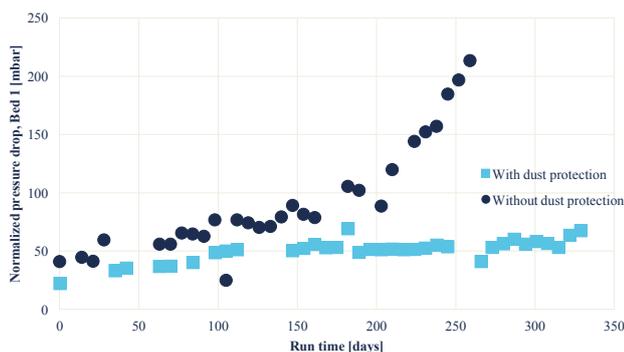


Figure 6. The effect on pressure drop buildup over the first pass of using dust protection catalyst.

the available room in the converter is now a bottleneck, both for lower emission and for further capacity increases.

Changing to more advanced catalyst

If the converter is full or if the temperature required to reach a high enough equilibrium conversion is too low for a standard catalyst, it is still possible to use a catalyst solution to achieve lower emissions. One solution is to replace some, or all, of the final bed with a more advanced cesium promoted catalyst. Due to its unmatched activity throughout the temperature range that is used in the last pass of a double absorption plant, Haldor Topsoe's cesium promoted VK69 offers a step change in performance in a plant. Figure 4 is an illustration of the conversion and emissions that are achievable at different catalyst loadings in the same plant as presented in the previous section.

As can be seen in Figure 4, a plant achieving 99.8% conversion in a 11.5% SO₂ feed gas with standard catalyst can boost its conversion to 99.9% by replacing the standard catalyst in the fourth bed with VK69. From Figure 4, it is also apparent that if there is enough available room in the converter and there are no temperature limitations in the plant, very high conversion is possible with this solution. The amount of VK69 in the last bed can be tailored to meet emissions legislations to take the specific limitations of the plant in question, as well as the performance of the other beds. Although the example presented in Figure 4 is for a plant with a 3+1 configuration, the trends will be similar for 2+1, 3+2, or 2+2 configurations as well.

The Haldor Topsoe LEAP5™ catalysts offer plant operators who already use cesium catalysts in the final bed another leap in performance towards higher conversion and lower emissions. Having the option of using a more active catalyst can help the operator avoid costly plant revamps and avoid cutting production rates if more stringent emission legislations

are introduced. It can also be used as a quick solution to compensate for lacking performance in other catalyst beds or parts of the plant. The LEAP5 catalyst can be employed either in the third bed of 3+1 or 3+2 plants, or in the last beds of single absorption plants. Figure 5 shows how LEAP5 catalyst can be used in an existing double absorption plant to achieve a higher conversion level.

Figure 5 suggests that the sulfur burning plant in the example could reduce its emissions by around 40% when using LEAP5 and VK69. This improvement is well in line with what has been seen in the industry. Operators installing the LEAP5 catalyst have seen an average emissions reduction of approximately 50% when replacing a used catalyst.

Maximising cycle length and fighting pressure drop

As with any other industrial chemical plant, minimising downtime and maximising on-stream time is critical for profitability. Although an effective maintenance plan is important for achieving this, the cycle length of many sulfuric acid plants will be dictated by the pressure drop over the plant in general and the catalyst in particular. The pressure drop typically builds up due to plugging of the first catalyst bed by deposition of dust.

As the dust deposits in the catalyst bed, the pressure drop will continue to increase. Finally, a point will be reached where either the blower cannot maintain design gas flow or the structural pressure drop limits of the SO₂ converter have been reached. At this point, the plant is typically shut down to allow for screening of one or more catalyst beds. This tends to result in a 10 day loss of production. Decreasing the effect of dust deposition and the pressure drop buildup rate is very beneficial.

Although decreasing the feed gas flow through use of higher feed gas strength will slightly improve campaign length by reducing the overall pressure drop, it will not address the underlying cause of the pressure drop problem – dust deposition. Reducing the amount of dust that enters with the feed gas would be possible through upgrading the sulfur and air filtration. However, this would require a substantial investment, which may be hard to justify financially. A solution that targets the dust issue directly, but does not require any substantial investment, is installing a layer of dust protection catalyst on the top of the first bed. This has the added advantage of being possible to install with very little preparation and it takes very little time to carry out. It could easily be implemented during the next shutdown.

Dust protection catalysts will not reduce the amount of dust coming in with the feed gas. Instead, they allow more dust to deposit in the bed before the pressure drop increases. The larger dust capacity is achieved through using larger catalyst pellets with larger void space to increase the penetration depth of the dust particles. Increasing the catalyst pellet size from 12 to 25 mm will increase the penetration depth by 100%, which also increases dust capacity and operating cycle length by the same amount.

Once the dust protection catalyst is installed in the client's plant, the operating cycle length is improved greatly. This saves the client significant sums in lost production and turnaround costs. A comparison between before and after the dust protection catalyst is installed is shown in Figure 6.

When comparing the data in Figure 6, it can be seen that the pressure drop increase operating with a dust protection catalyst for 325 days is approximately the same as when operating without it for around 150 days. Although the plant had not yet shut down when the data was taken, based on these trends, it can be expected that the improvement in campaign length is close to the 100% that theoretical calculations indicate. Over a 10 year period, a longer campaign length would result in a decrease in the number of necessary shutdowns for catalyst screening from 13 to six. Assuming that 10 days are required for a shutdown to screen bed one, an additional 70 days of production will be achieved. Furthermore, each shutdown that can be avoided will result in further savings in costs for screening contractors, make-up catalyst and fuel for startup and purge.

Using catalyst to reduce energy costs

The cycle length of many sulfuric acid plants is limited by the pressure drop buildup over the catalyst and can fully utilise the larger dust capacity to achieve increased campaign length. However, this is not the case for all plants. Other plants may have different bottlenecks that make a longer campaign length impossible, but even these plants may benefit from installing a dust protection catalyst to achieve a lower average pressure drop over the plant. To demonstrate how such a plant could benefit from using a layer of dust protection catalyst, an example is set up below, based on the industrial data shown in Figure 6. Energy savings that are achieved by a lower pressure drop can be calculated with the following formula:

$$Q = \frac{G \cdot \Delta p \cdot 0.024}{0.7}$$

Where Q is the difference in energy consumption per year (kWh), G is the flowrate (Nm³/h) and Δp is the pressure drop difference (mm Wc). If the data pressure drop formula above is applied to the example plant, the yearly savings in terms of power usage can be calculated to 4 GWh. With an average electricity price in the EU of €0.119/kWh. This corresponds to annual savings of approximately €475 000.

An important side effect of adding a dust protection catalyst to reduce the average pressure drop over the campaign is added dust capacity. Added dust capacity will reduce the risk of the plant having to shut down prematurely due to a higher than normal dust load. In such an event, the dust protection will yield similar savings as presented for the example plant.

Conclusion

Modern sulfuric acid production technology is more or less synonymous with contact plants. Depending on the SO₂ source, these plants could operate under either dry conditions or wet conditions, such as Haldor Topsoe's WSA process.

Being at the heart of the sulfuric acid plant, the SO₂ oxidation catalyst will have a profound effect on how a plant performs. With modern SO₂ oxidation catalysts, such as VK69 and LEAP5, very low SO₂ emission can be achieved. The effect of choosing the right catalyst solutions is not limited to emission control, but can also help to improve capacity, cycle length and decrease pressure drop. **WF**



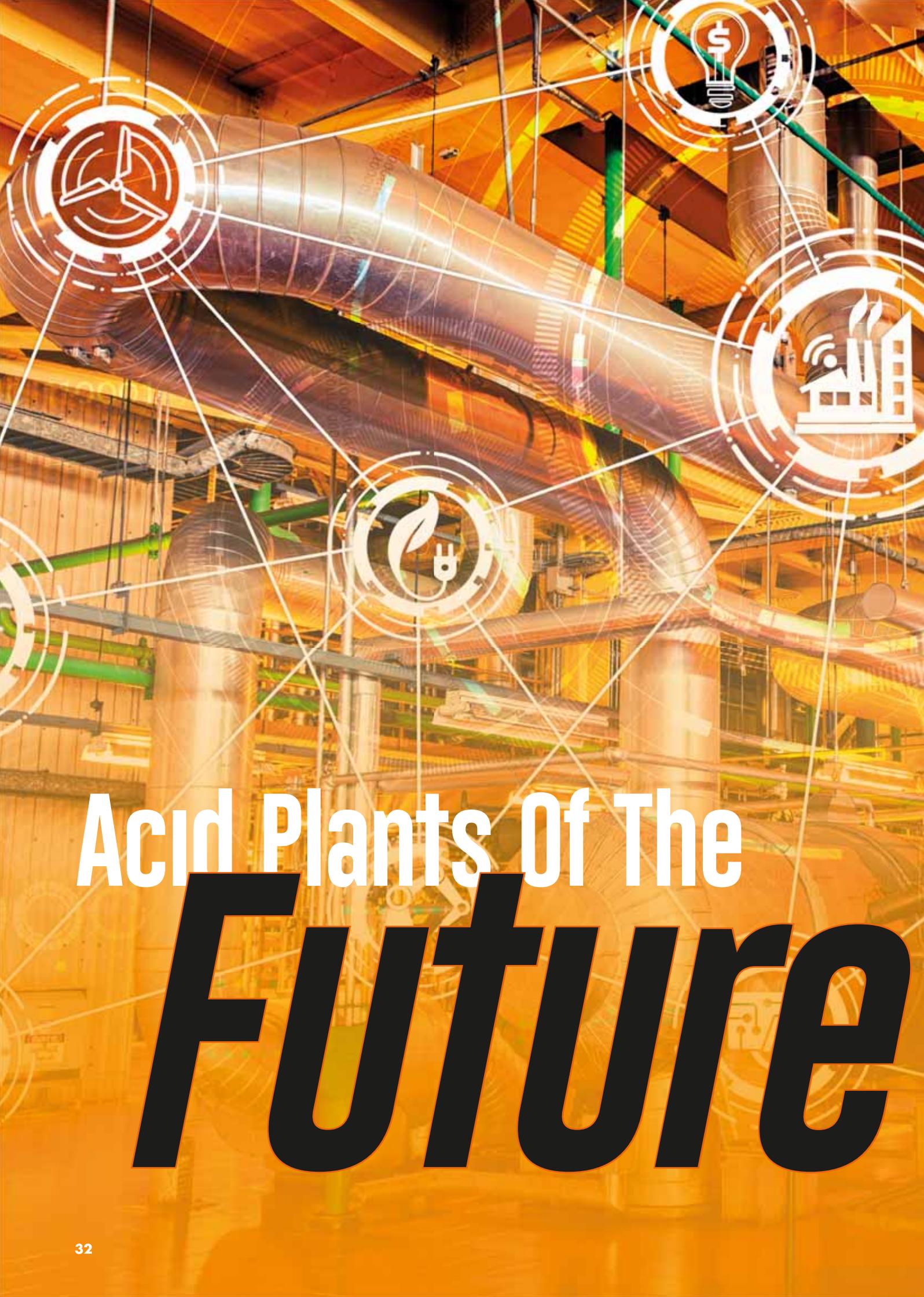
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In any phosphate fertilizer complex, the sulfuric acid plant plays a critical role. This is because it provides not only the sulfuric acid that is needed in the phosphoric acid plant, and potentially the fertilizer granulation plant, it also provides the electrical power and steam that is required for other plants at the complex. If the sulfuric acid plant is not able to operate reliably at desired operating rates, this can result in difficult or impossible operation of the linked phosphoric acid, power and fertilizer granulation plants. Having all of the plants on site running consistently at design capacity for long periods and without unplanned stoppages is the fastest way for operators to maximise return on their investment.

Sulfuric acid plants deal with aggressive chemicals, including sulfur dioxide (SO_2) and sulfuric acid (H_2SO_4), which puts a lot of stress on the plant equipment. Operators of sulfuric acid plants are thus continually trying to find more suitable technology that is characterised by the following criteria:

- A capability to comply with and often exceed all statutory environmental regulations.
- Self-evident potential for low maintenance and high reliability.
- A capability to achieve enhanced energy use.
- Cost-competitive and safe to operate and maintain.
- Simplified and innovative designs that allow simple installations or retrofit.

In response to these challenges, Chemetics has been working to eliminate many of the persistent deficiencies with enhanced process and mechanical equipment design.

Sulfuric acid plants are considered as a source of energy for steam production and electric power generation. Extracting more of the energy that is released during the production of H_2SO_4 is increasingly important to create additional revenue for plant operators and so they can have less reliance on external power grids for energy.

In order to achieve up to 99% energy recovery from the sulfuric acid process, Chemetics has developed a family of process add-on systems that recover additional energy without sacrificing plant availability.

Equipment designs

To eliminate the recurring maintenance problems that are associated with traditional sulfuric acid plant designs, the company has been working on recent innovations that allow operators to minimise the

amount of work that needs to be done in a hazardous environment, while also reducing the installation and maintenance time and costs.

Modular converter

The converter is at the heart of the sulfuric acid process. It is the vessel containing the series of catalyst beds that are required to convert SO_2 to sulfur trioxide (SO_3). In earlier days, this vessel was manufactured from partially brick-lined carbon steel and cast iron. Although many hundreds of these conventional units are installed around the world and are still in use today, most have caused the owners significant operating and maintenance problems. Changing the construction material to stainless steel and using an all welded design has resulted in the elimination of many maintenance issues.

Chemetics' converter design can further incorporate one or two internal gas exchangers and/or superheaters inside the core of the converter. This eliminates the hot gas ducting between beds 1 and 2, which is well known as a continual maintenance problem for many plants due to very high gas and metal temperatures (Figure 1).

Typically, converters that are too large to be shipped fully fabricated are built piece by piece on-site. On-site fabrication has substantial risks associated with it due to the concurrent risks of inclement weather conditions, proximity to operating equipment, as well as the lack of available skilled welders and fitters local to the plant site.

Since the early 2000s, Chemetics has switched to supply virtually all of its stainless converters in a modular form to minimise field construction. The converter is shipped to the site as prefabricated modules before they are assembled on-site. This method reduces construction time by approximately 70 – 85% and improves the overall quality of construction as the majority of the welding and fitting is completed off-site in a fabrication shop under ideal conditions (Figure 2).

Acid towers

Silicon-containing stainless steel, such as SARAME[®], in hot strong sulfuric acid service have been replacing the traditional standard of ductile iron acid piping and, more recently, in certain acid tower and acid cooler applications. In retrofit situations, when the shutdown time is short, using alloy acid towers to replace existing brick-lined acid towers can be an attractive solution. Alloy towers have the advantage of allowing for the prefabrication of either the entire tower or the tower

modules, including internals off-site. The use of alloy towers eliminates the premium cost and additional brick settling time required when using specialist contractors to complete the acid bricking installation on-site.

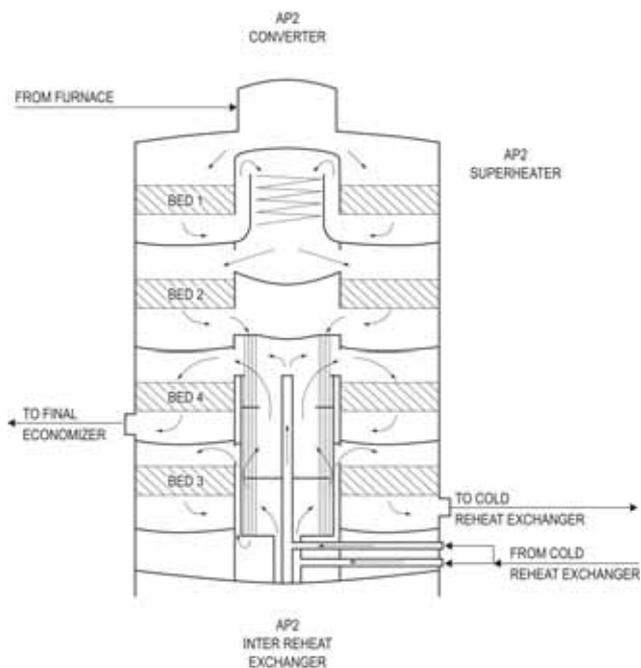


Figure 1. A converter with an internal superheater and gas exchanger.



Figure 2. Converter modules being assembled in-field.



Figure 3. A complete SARAMET alloy acid tower being lifted into position.

Acid distributors

The acid distributor in the acid tower provides uniform distribution of acid into the packing section. Common issues such as blocked downcomer tubes, unequal flow due to gas channelling, and/or uneven level of distributors can lead to poor SO₃ and water absorption. Ultimately, this leads to accelerated corrosion in downstream equipment or high stack emissions.

Chemetics fabricates its acid distributors from SARAMET austenitic stainless steel, which has corrosion and erosion resistant properties in strong sulfuric acid service. The distributors provide uniform acid tower irrigation thanks to a high downcomer tube density, a stable acid level inside the trough due to a calming plate design, and individual tube orifice flow control.

The ISO-FLOW™ construction is not sensitive to acid inlet velocities or trough levelling. It is, therefore, more forgiving than overflow weir style designs. Calming plates incorporate filter screens that are easy to clean and prevent any debris from entering the acid distribution tubes making this design resistant to suspended solids, such as packing chips and mortar pieces. The internal filters prevent the plugging of downcomers during normal operation.

Improvement in the ease of assembly to reduce the shutdown time required for installation can be achieved in the SWIFTLOCK™ design by eliminating field installation hardware and reducing the site assembly parts to as few as possible.

Segmental tube in gas exchangers

As the size of acid plants continues to increase, demand for larger gas-gas heat exchangers continues concurrently. Even though a modern gas exchanger is significantly more efficient than a traditional design, they require very large exchangers, which can weigh in excess of 200 t. This approach of field fabrication results in many or all of the critical tube to tubesheet welds being done in the field in non-ideal environmental conditions. Additionally, the time required for fabrication and the lack of skilled trades for work on-site makes the cost often prohibitive.

To overcome the issues of fabricating large gas exchangers, Chemetics has developed a method where the tube bundle is modularised into segments that can be assembled easily and safely on-site. With this methodology, approximately 85% of the gas exchanger assembly time and over 90% of the critical welding is done in a shop environment. The remaining welding and fitting can be accomplished by a small team of experienced fitters and welders on-site.

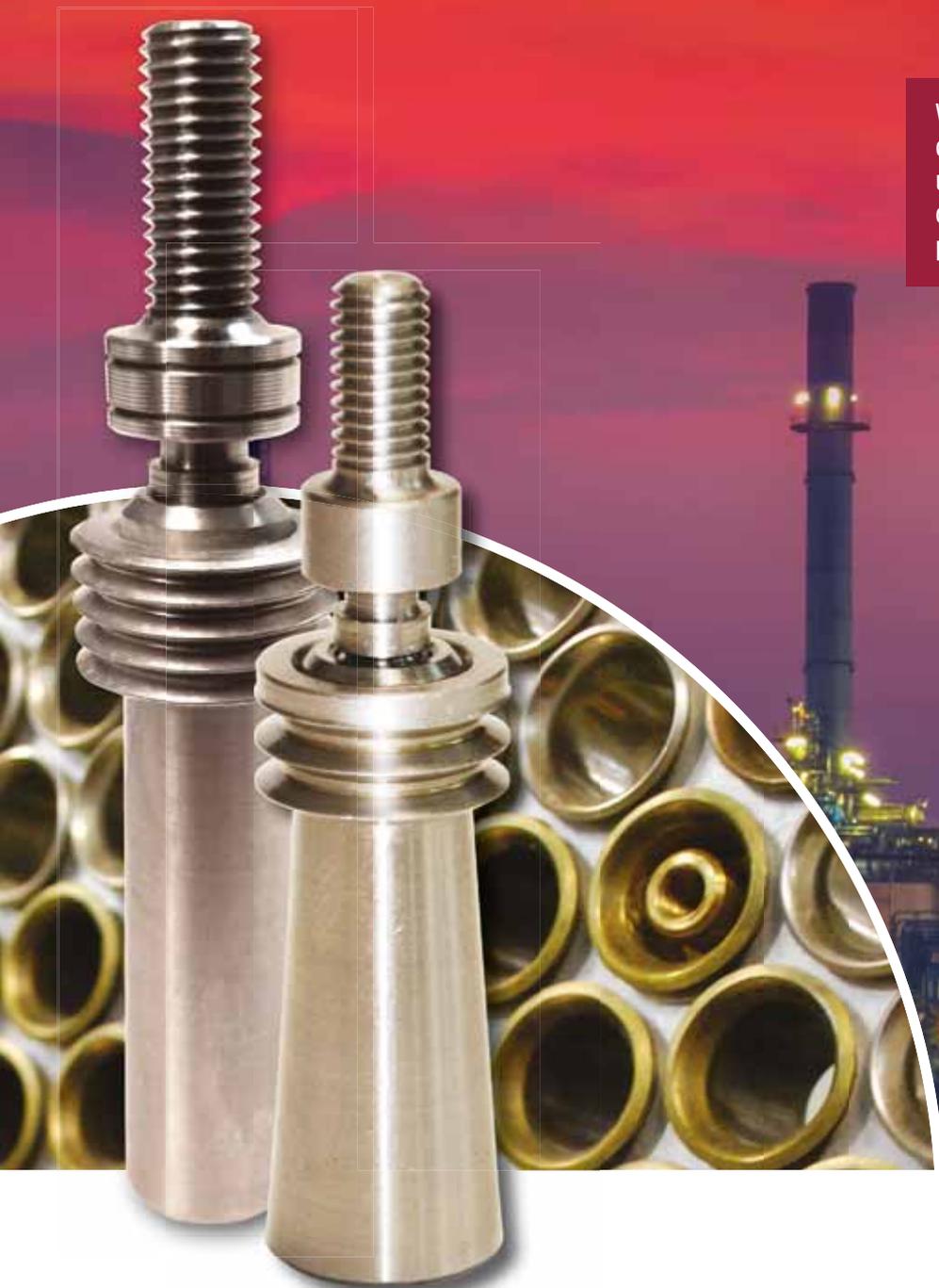
Combining a gas exchanger and catalyst

A conventional acid plant is limited to 12 – 13% SO₂ at the converter inlet to keep the gas temperature from leaving the first pass of the catalyst bed below the thermal stability of vanadium-based catalyst (approximately 630°C). To counteract this limitation, Chemetics offers Cooled Oxidation Reaction (CORE®) converter technology – a pseudo-isothermal reactor system that can convert high strength SO₂ gas without diluting the gas with air or recycling process gas. Its first commercial installation was in

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Germany, where it has been operating continuously for more than eight years. A second larger plant was commissioned in February 2017 and passed its performance test in April 2017 (Figure 5).

This converter system for SO₂ oxidation uses conventional vanadium-based catalysts with SO₂ gas flows through the tubes.



Figure 4. Modular gas-gas heat exchanger.



Figure 5. The CORE® add-on installation.

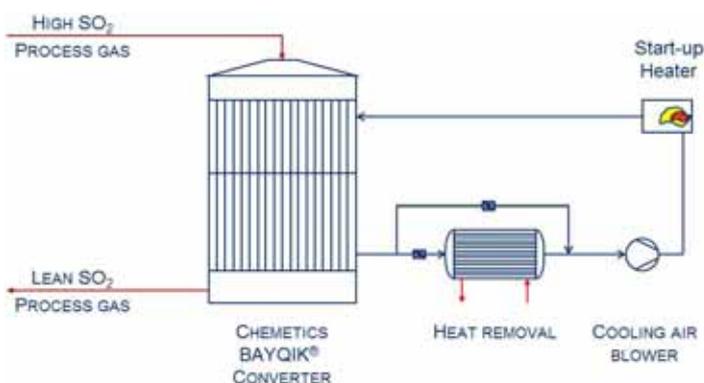


Figure 6. The CORE system.

Continuous removal of reaction heat on the shell side allows the process temperature to be controlled within the operating limit of the catalyst. Energy recovered from the heat removal system can be used for preheating the process gas and generating high pressure steam (Figure 6). The pseudo-isothermal process operates further away from the equilibrium curve than a traditional multi-pass adiabatic process, as shown in Figure 7. This results in lower overall catalyst loading and significantly higher SO₂ conversion in a single pass.

Energy recovery

Traditionally, in sulfuric acid plants, only the high grade energy from the hot process gas is recovered as this can be used to generate high pressure (superheated) steam, which can then be used as a heat or power source in the complex. In recent years, lower grade energy from the liquid streams is increasingly being looked at to provide additional revenue for plant operators. The main source of low grade energy at temperatures of 100 – 200°C, is the absorption energy, released when the acid leaves the intermediate absorption tower with much smaller amounts available from other streams.

The Chemetics Acid Low Pressure Heat from Absorption (ALPHA™) system produces valuable steam from the energy that is released during the absorption of SO₃ into strong acid in the tower. Steam is produced at pressures up to 10 Barg. This add-on system with a separate hot absorption tower allows the acid plant to remain fully operational, even when the system is not in use. One of the main benefits of this design is that it can be stopped or started independent of the rest of the sulfuric acid plant, allowing for easier and more flexible plant operation.

Another solution for extracting energy from acid streams is the CES-HWS™ (Hot Water System). This produces hot water from the acid in the absorber circuits. For this process, the anodically protected acid coolers in the absorption circuit are specially designed to allow significantly higher water side temperatures. If higher water temperatures than approximately 105 – 110°C are required, they can be reached using SARAMET coolers. To benefit from hot water production, it is necessary to have a user within reasonable distance that can use this hot water.

The CES-DSW™ system, or Desalinated Sea Water, is a further evolution of the CES-HWS. Hot water is generated in the acid plant, but it is used in a closed loop to produce desalinated water from sea water or brackish water using a multiple effect distillation (MED) system. Generated at 90 – 95°C, the hot water flows from the acid plant to the MED unit where it is used in sea water heaters to create low pressure (0.5 bara) flash steam (Figure 9). This flash steam is, in turn, the energy source for the MED to produce desalinated water. Depending on the efficiency of the MED unit, up to 10 kg of desalinated water can be produced from each kilogram of steam that enters the unit.

It is easily understood that the combination of a sulfuric acid plant together with a multiple desalination unit is a very economical way to produce desalinated water in areas where fresh water is not readily available. Compared to sea water reverse osmosis, the MED provides better quality water, has fewer operational issues and lower production cost. Operation of the MED system is easy and requires minimal attention from the operator.

The following should be considered when evaluating the preceding options:

- Producing hot water is significantly less expensive than producing steam. It also recovers a greater percentage of the energy.
- If a useful outlet can be found for hot water, then this is the most cost-effective and most reliable solution.
- For locations where fresh water is scarce and desalinated water is required, the CES-DSW system is often one of the lowest cost solutions to produce desalinated water.

For locations where no outlet for hot water is available, the production of steam and/or power should be considered. In this case, the client should select a technology that provides steam at the lowest cost and the lowest risk to their main process. The CES-ALPHA, which can be completely bypassed if required while maintaining full acid and steam production, is a good example of such a system.

Conclusion

A number of options are available for recovering energy from acid plant absorption circuits, including low pressure steam generation, hot water generation and desalination water generation. With hot water and desalination water generations options, around 7% more heat can be captured while comparing with the low pressure steam generation option. These options have different cost implications and advantages. In order to determine the best solution for the site, it is important to utilise a technology provider that can offer a variety of options.

Sulfuric acid plants with high performance equipment can be operated for extended periods of time, i.e. higher than 98.5% plant availability. End-users of Chemetics' higher performance

equipment benefitted with lower CAPEX, lower emissions, ease of operation with lower maintenance, higher reliability and acid plant commissioning time of less than three weeks to achieve the designed production rate. **WF**

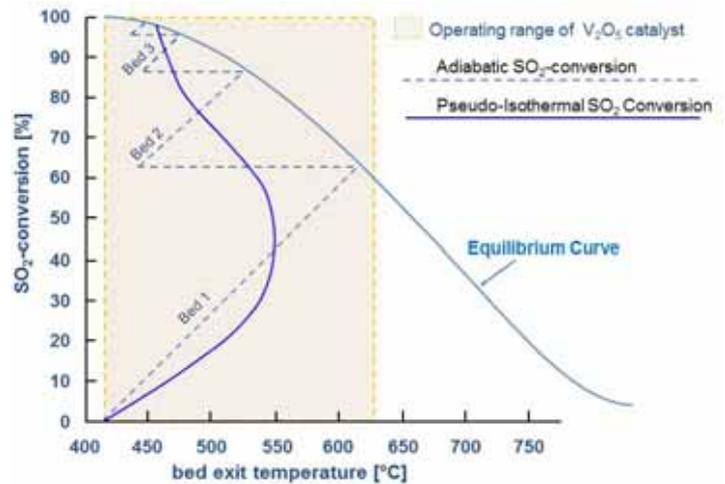


Figure 7. SO₂ conversion curve of conventional adiabatic process vs pseudo-isothermal process.

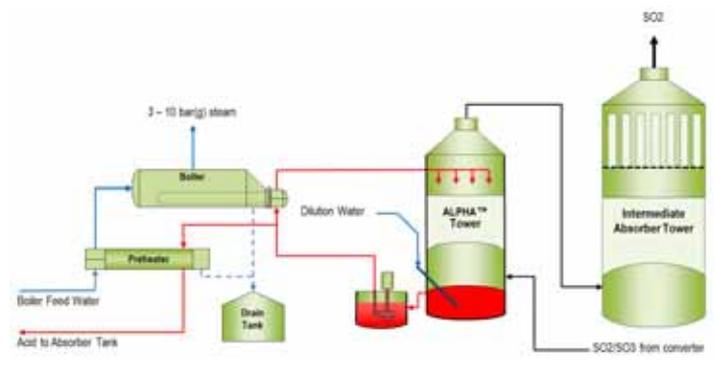


Figure 8. A schematic flow diagram of the CES-ALPHA system.

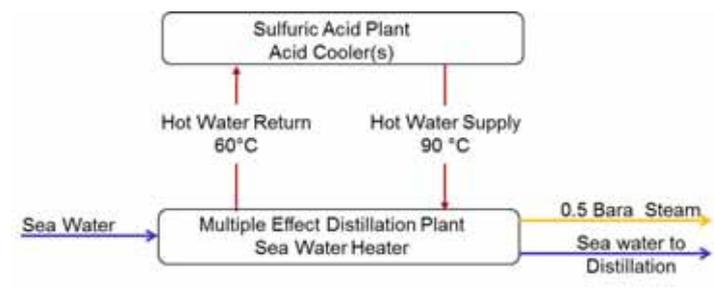


Figure 9. Integration of a sulfuric acid plant with a MED unit.



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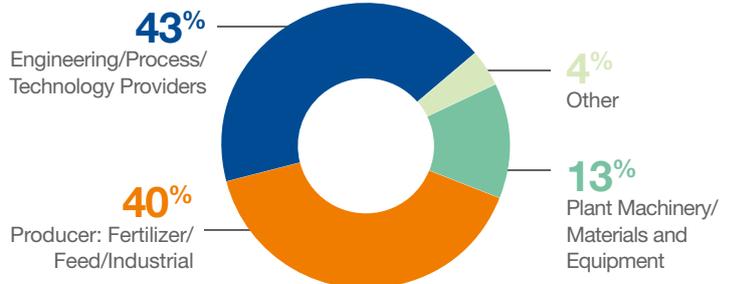
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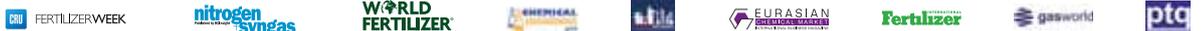
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SULFURIC ACID GAS CLEANING

Gary Siegel, Beltran Technologies, Inc., USA, explains the effectiveness of simple modern electrostatic precipitator designs.

Industrial grade sulfuric acid is still the most widely used industrial chemical in the world. It continues to be sourced primarily as a non-discretionary byproduct from the roasting, smelting and refining of non-ferrous metals (70%), as well as from natural gas processing, electric power generation, and spent acid regeneration. These industries tend to be heavy emitters of particulates, sulfur and nitrogen oxide gases, and sulfuric acid mists, amongst other pollutants. They are also subject to increasingly strict environmental regulations.

When concentrations of sulfur dioxide from these operations exceed 5 – 7% of exhaust gas volumes, a common and cost-effective solution is incorporating a downstream sulfuric acid manufacturing plant. Owners of these facilities can capitalise on the high industrial market value of purified sulfuric acid, while achieving greater operating efficiencies and easier regulatory compliance.

However, an efficient sulfuric acid manufacturing process requires as much removal of fine particulates, acid mists,

condensable organic compounds and other contaminants from input gas streams as possible. This high level of gas cleaning efficiency is necessary to prevent poisoning of the catalysts, as well as fouling or plugging of the catalyst beds. An optically pure input gas is essential for avoiding the formation of a 'black' or contaminated acid end product. Proper gas cleaning also helps protect sensitive acid plant components against corrosion damage, thus lowering long-term expenditures for maintenance and equipment replacement. Ultimately, cleaner gas streams facilitate the production of stronger concentrations of sulfuric acid that is suitable for a wider range of end uses.

Wet electrostatic precipitators

Modern gas cleaning and emission control strategies continue to evolve in response to the increasing complexity, toxicity and corrosiveness of industrial exhaust and process gases – not to mention increasingly stringent environmental regulations. Sulfuric acid plant owners have employed several gas cleaning techniques,



Figure 1. Beltran WESPs installation at Hindustan Zinc Ltd.

including wet or dry scrubbers, cyclones and fabric filters. These systems can capture larger particulates, but are usually energy-inefficient or impractical to use on fine particulates, acid mists, oily residues or condensed organic compounds. Therefore, many plant operators find that the best solution for removing these contaminants is a modern adaptation of a traditional technology: the electrostatic precipitator (ESP).

The basic ESP design features an array of discharge electrodes, such as negatively charged wires or rods, which can generate a strong corona field. Each electrode in the array is surrounded by a positively charged or grounded collection surface, traditionally the interior of a square or cylindrical tube. In operation, the source gas is passed through the array as the ionising electrodes induce a negative charge in even the most minute, sub-micron sized particles. These are instantly propelled towards the interior collection surfaces, where they adhere as the cleaned gas is passed through.

This basic design concept offers several inherent advantages, the primary one being the ability to capture fine, sub-micron sized particulates and acid mist droplets, whose minimal mass enables them to escape through scrubbers and other mechanical equipment.

Another advantage that is inherent to precipitators in general is the extremely low pressure drop that is experienced as gases pass through the system, which can reach as low as 1 – 2 kPa. With virtually no mechanical impedance or obstruction of target pollutant particles travelling through the ESP, both gas velocities and operating efficiency can be extremely high. This enables plant designers to use smaller-scale, less costly equipment and still achieve high quality collection efficiencies compared to other systems.

For engineers and plant designers in the field of gas cleaning, several operational goals stand out:

- Achieving the highest level of particle collection efficiency.
- Cleaning greater volumes of source gases, with faster throughput speeds.
- Achieving the greatest reductions in costs related to capital investment, operating costs, energy consumption, equipment maintenance and long-term equipment replacement.

To help sulfuric acid plants achieve these goals, Beltran Technologies, Inc. has been researching and developing a

specific type of advanced precipitator technology that offers proven high performance and efficiency – the wet electrostatic precipitator (WESP). WESPs can vary greatly in their design, materials, gas flow rates and durability, as well as collection efficiency. As a result, it is important for engineers to recognise the key differences among these various systems.

Primarily targeted at capturing sub-micron scale particulate matter, saturated sulfuric, or other acid aerosols and condensable organic chemicals, Beltran Technologies' WESP uses aqueous flushing nozzles to clean the collection surfaces. The particles that are captured are cleansed from the collection surfaces by recirculating water sprays. Residues, including aqueous sulfuric acid, are also extracted for further use or disposal. The cleaned gas is then ducted to downstream equipment or to the stack, depending on the application. A well-designed and correctly-operated WESP can clean complex gaseous emissions of particulates and acid mists down to sub-micron scale (PM 2.5) with up to 99.9% efficiency, and very low energy drain.

The WESP concept is basic, which makes it versatile for use across a range of industries, applications, operating conditions and gas chemistries. This adaptability is critical to metallurgical and petroleum refining operations, where source gases can be highly variable.

Taking advantage of the inherently low pressure drop of ESPs, Beltran Technologies' staff devised a multistage system of ionising rods bristling with star-shaped discharge points. These are encased within square or hexagonal tubes to maximise the collection surface area and minimise overall space requirements. The unique electrode geometry generates a corona field that is approximately four or five times more intense than that of conventional wet or dry ESPs resulting in greater particle migration velocity and adhesivity. The multistage charging configuration also avoids corona quenching due to high particle densities, and assures maximum corona field strength with a minimum of energy load.

Overcoming challenges

A persistent challenge facing traditional dry-operating precipitators is the re-entrainment of particles from collection surfaces back into the gas stream due to the use of mechanical or acoustical rapping units. Beltran Technologies' design uses a vertical flow and continuous aqueous flushing of WESPs to significantly minimise this problem. By eliminating the need for rappers, WESPs also reduce both the higher cost and energy drain imposed by that equipment. For industries that generate oily or sticky residues, the aqueous flushing also prevents particle build-up, overcomes electrostatic resistivity and back-sparking on the WESP collection surfaces.

Other critical features to look for in WESP equipment are sophisticated electronic controls that are linked to a close-coupled gas flow management system. These can optimise operating parameters, such as gas velocity, saturation, temperature and corona intensity, to achieve maximum efficiency.

A major threat to the cost-effectiveness of a WESP is equipment corrosion caused by the harsh chemical components of treated gases. To prevent premature deterioration, critical surfaces are constructed with advanced protective materials, such as fibre-reinforced plastics or high nickel-chromium alloys. The high voltage insulators are continuously purge-air cleaned to further reduce maintenance costs. **WF**



THE PURSUIT OF PERFORMANCE

Douglas Azwell, MECS, USA, explores the impact of mist eliminators on sulfuric acid tower performance.

Proper performance of absorption towers in sulfuric acid plants is a critical step in the path towards optimum plant performance. Much time and effort is expended, and rightly so, on inlet gas temperature, inlet acid temperature, acid concentration, acid flowrate, distributor type, packing type, packing depth, etc. However, there is another piece of equipment that is key to the proper operation of towers: mist eliminators. Choosing the correct mist eliminator type and style can make a tremendous

difference to tower performance, affecting not only stack appearance. It can also make a huge difference in terms of maintenance of downstream equipment, ductwork and catalyst as years of operation pass.

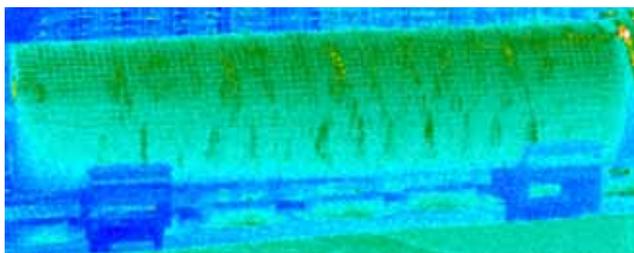


Figure 1. A MECS ES angle wrapped mist eliminator.

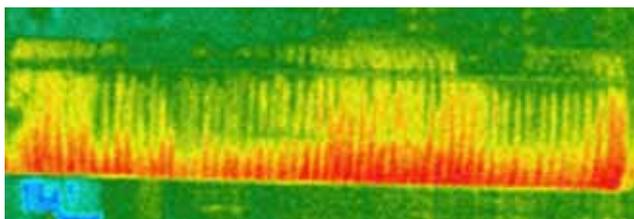


Figure 2. A parallel wrapped mist eliminator.

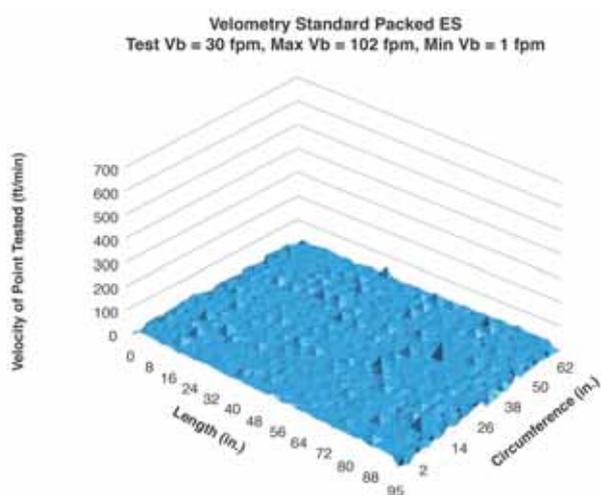


Figure 3. 3D velometry of an angle wrapped mist eliminator.

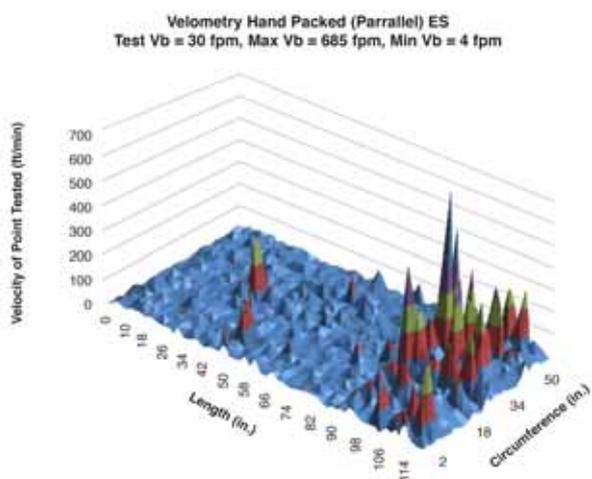


Figure 4. 3D velometry of a parallel wrapped mist eliminator.

There are a number of mist eliminator options on the market that are intended to capture liquid droplets in sulfuric acid plants, but they do not all function in the same way. These devices rely on three different mechanisms for capturing mist: impaction, interception and diffusion. MECS® mist eliminators use all three of these mechanisms to achieve optimum performance. The impaction mechanism is used predominantly with impaction devices (such as mesh pads and CS™ candles), the interception method is used in a variety of systems, while the diffusion mechanism is featured in the Brownian diffusion devices. These mist eliminator devices are frequently installed in sulfuric acid plants for long-term, regulatory compliant and reliable operations.

Brownian diffusion fibre beds collect a variety of droplets in process gases, ranging from less than 1 µm to those that are substantially larger than 3 µm. These devices are able to collect the droplets efficiently by means of the three previously mentioned mechanisms.

The focus of this article is on diffusion bed mist eliminators, which are predominantly used in interpass absorption tower (IPAT) and final absorption tower (FAT) installations in modern acid plants. However, notably, there are some plants that operate impaction/interception type mist eliminators in various parts.

Brownian diffusion bed mist eliminators are produced by either:

- Compressing bulk glass fibre between two acid-resistant metal cages (as is the case with the HE™ or high efficiency brink mist eliminator).
- Wrapping glass fibre (in rope form) around an inner cage that is constructed of acid-resistant metals (as is the case with the ES™ type or energy saver brink mist eliminator).

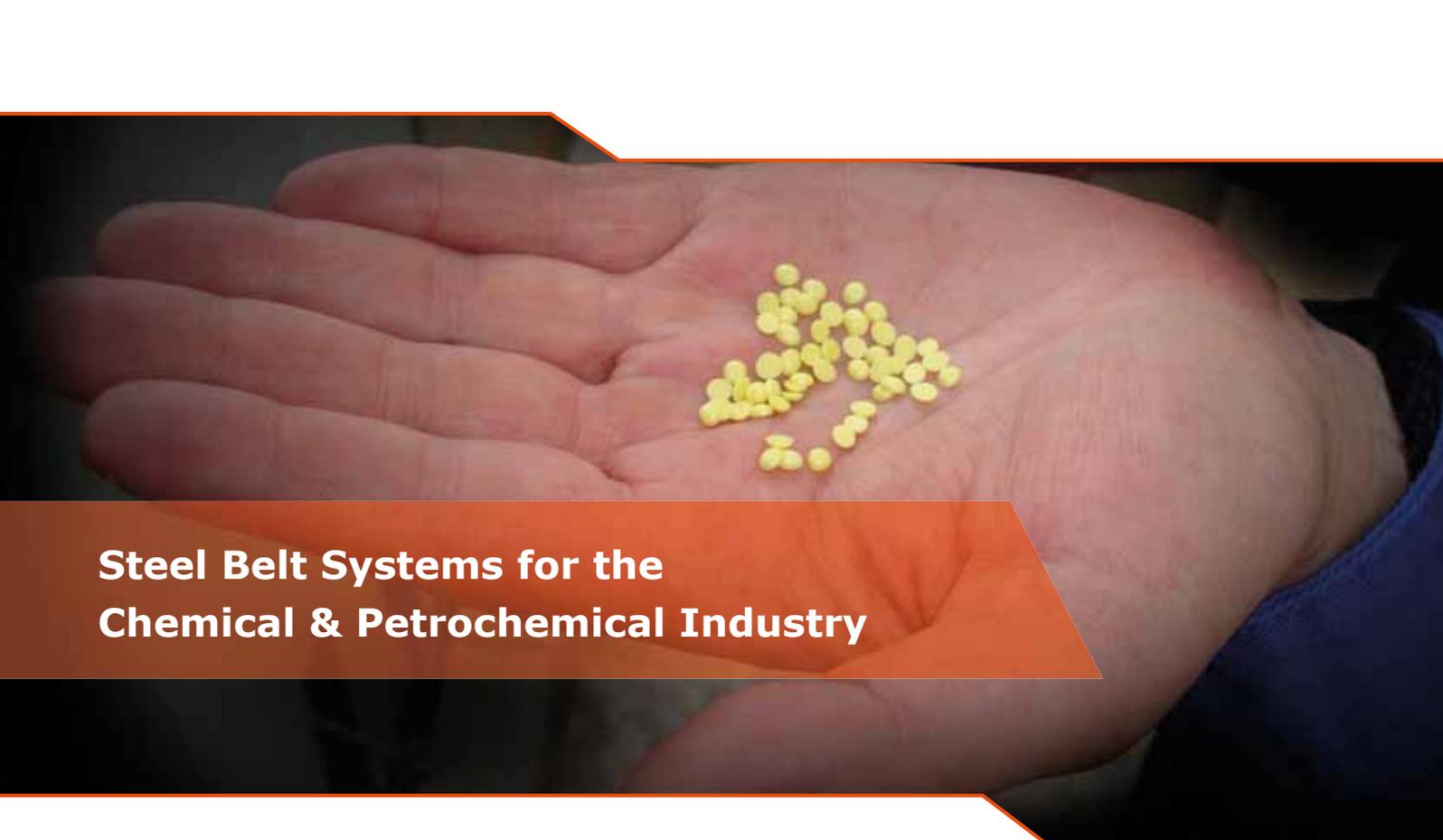
Angle wrapping

In the case of the rope form of fibre placement, which MECS calls the ES type, the positioning of fibre has a significant impact on the efficiency of the final product. If there are gaps between the fibre ropes, there is a path of least resistance for the gas to follow, which entails diminished efficiency. The effect of fibre placement on performance is easily demonstrated if a thermal camera is used to produce an image of a mist eliminator with gas flow, as demonstrated in Figures 1 and 2. Angle wrapped mist eliminators show fewer areas of gapping, while the gap between every rope can be easily seen in a parallel rope packed bed.

While thermographic imaging provides a qualitative measure of a mist eliminator's quality, it is possible to quantify the performance with a device to measure bed velocity over the surface of a fibre bed. Velometry testing of angle wrapped and parallel wrapped mist eliminators shows that the packing style used plays a significant role in overall performance of a mist eliminator. As shown in Table 1, parallel packed beds have velocity excursions that are over six times the worst case compared to angle wrapped ES fibrebeds. These excursions contribute to mist penetration and reduced efficiency.

In addition, a 3D representation of a fibre bed's velocity profile can be produced from this data, illuminating problem areas on the mist eliminator. Figures 3 and 4 demonstrate the visualisation potential of the 3D graph generated from using data that was taken during velometry testing.

Most of the mist carryover from a mist eliminator that has a velocity profile similar to Figure 4 would be in the form of large 5 – 10 µm re-entrainment. These droplet sizes would lead to



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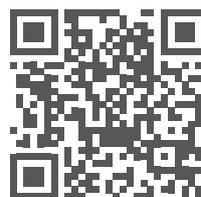


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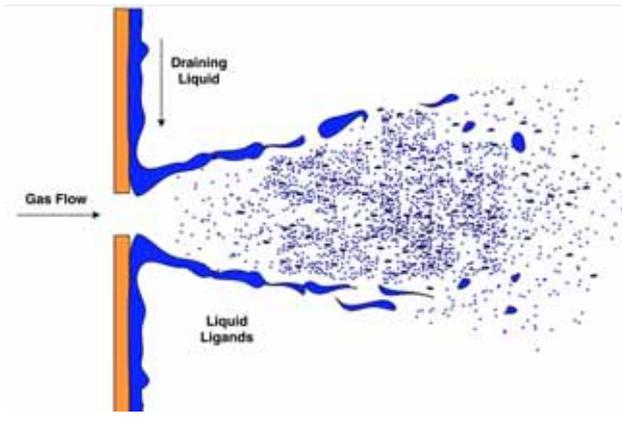


Figure 5. Depiction of re-entrainment.

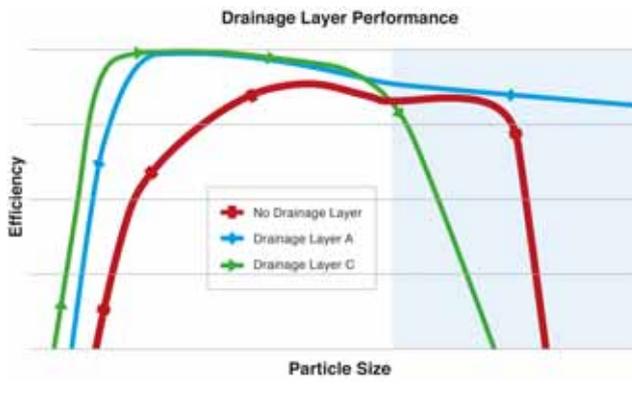


Figure 6. Laboratory test results for drainage layer optimisation.

Table 1. Velometry data of the ES mist eliminator packing style

Mist eliminator type	Test bed velocity (FPM)	Maximum bed velocity (FPM)	Minimum bed velocity (FPM)
Standard packed (angle wrapped)	30	102	1
Hand packed (parallel wrapped)	30	685	4

downstream ductwork and heat exchanger corrosion if, after an IPAT, or in the case of a FAT installation, the acid would drain from the stack.

Re-entrainment occurs when a portion of the captured mist draining by gravity from the fibre bed is regenerated into mist particles that are carried back into the gas stream, eventually resulting in downstream stack emissions and/or equipment damage. This phenomenon can occur when the gas velocity through the mist eliminator is sufficiently high so as to cause interaction with draining liquid from the fibre bed. This results in liquid particle separation. Thus, high velocity spikes in a fibre bed will, necessarily, lead to zones of high re-entrainment and high mist carryover downstream in the process, as depicted in Figure 5.

Drainage

While angle wrapping is a highly effective means of reducing re-entrainment (by preventing velocity spikes), proper drainage layer design is another effective technique that can be employed to reduce re-entrainment and improve overall mist eliminator

performance. A bi-component fibre bed with specially-engineered collecting and drainage media allows collected liquid particles to find open pathways in the drainage media and to drain by gravity to the bottom of the element.

Though the concept itself is relatively simple, it is important to note that drainage layer design is a complex science that involves intricate interplay between fibre diameter, density, permeability and more. When properly balanced, these complex relationships can be leveraged to optimise element drainage and ultimately reduce re-entrainment. When improperly engineered, drainage will be negatively impacted and re-entrainment will occur.

Laboratory testing that has been conducted by MECS illustrates this concept. The tests show that drainage layer performance can vary widely, and that properly engineered drainage layers are a complex and crucial component of proper element design.

The blue line in Figure 6 would be a good choice for minimising re-entrainment because its overall efficiency (as shown on the Y axis) is higher than many other designs, particularly for larger particle sizes (as shown on the X axis). This is indicative of low re-entrainment. By contrast, the green line in Figure 6 would be a poor choice for minimising re-entrainment as its efficiency for larger particle sizes is worse than using no drainage layer at all.

As re-entrainment has the potential to generate relatively large mist particles, minimising its occurrence is of the utmost importance. Both angle wrapping and drainage layer design have important roles to play. When done correctly, mist carryover is minimised, emissions are low and downstream equipment damage can be avoided. When done poorly, the results can be reasonably costly for owners and operators.

Proper sealing

As well as the type of mist eliminator chosen and the design of the element itself, the method of attachment to a tower's tubesheet also plays a role in the effectiveness of the installation. Sulfuric acid producers must be keenly aware that the gasket, hardware torque, attachment style (direct attachment or pedestal style), seal leg condition and the process all play a part in the overall operation of the tower. If issues such as excessive drain rate or stack tail with the installation are identified, other potential problems, such as acid vapour/sulfur trioxide carryover, acid strength, acid circulation, inlet gas temperature, distribution issues and a myriad of other items, must be sorted out first in order to determine whether the issue has been caused by the mist eliminators themselves.

Conclusion

In the case of mist eliminators, it is what is inside that counts. While many designs look similar, details such as angle wrapping vs parallel wrapping and drainage layer design can have significant impacts on element performance. For mist eliminators to perform properly, details and experience matter. When managed properly, the results can be very beneficial for owners and operators, resulting in well-performing towers, improved mist capture, low emissions, reduced maintenance costs and low pressure drop.

However, as the test results in Figure 6 demonstrate, such achievements in mist eliminator technology are not a simple matter, and there is a great depth to mist eliminator science. Fortunately, the science has come a long way and through a full understanding of that science it is possible to leverage the latest industry technologies in a way that allows owners and operators to manage harsh process environments and meet tough design and regulatory requirements. **WF**



A STORY OF PROGRESS

Filippo Colucci, Solex Thermal Science Inc., the Netherlands, compares conventional fluid bed coolers and indirect plate coolers, with a focus on energy consumption.

Fertilizer processing plants have continually been under pressure to revamp operations in order to remain competitive in today's market. They are also dealing with tight operating margins and the need to comply with current and new environmental regulations.

Just over 20 years ago, a new technology was introduced to the fertilizer industry by Solex Thermal Science Inc. – an indirect plate heat exchanger that was used as the final cooling step before the product was stored. This method of cooling provided an alternative to the traditional fluid bed cooler or drum cooler, which was the previous industry standard. It offered several key advantages compared to the fluid bed cooler, including lower energy consumption, lower air volumes, and a smaller footprint. There were also savings in installed capital cost by eliminating the large air handling system for the air coolers, which were needed for the fluid bed cooler.

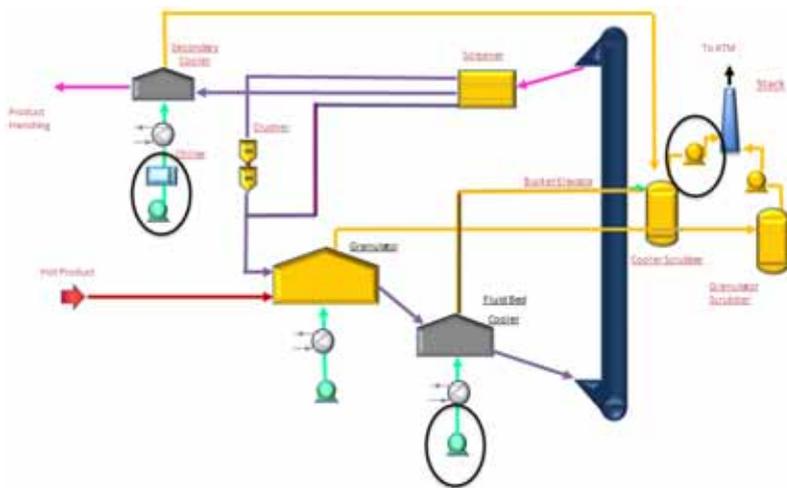


Figure 1. The typical granulation process using a fluid bed cooler.

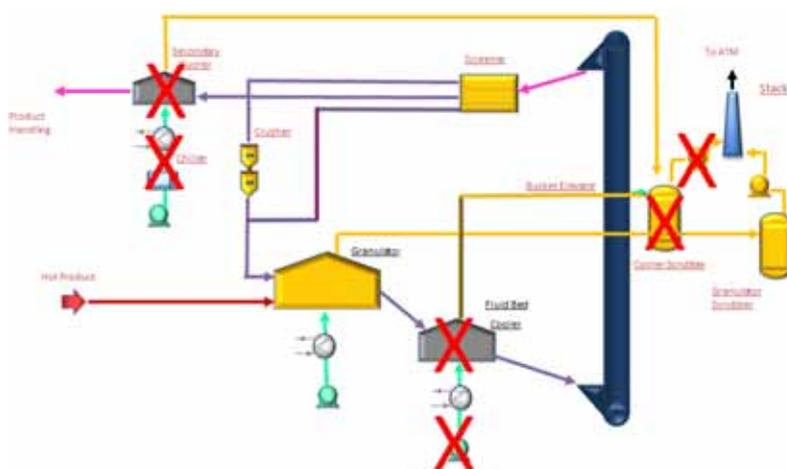


Figure 2. Changing to an indirect plate cooler eliminates the need for several pieces of equipment associated with a fluid bed cooler.

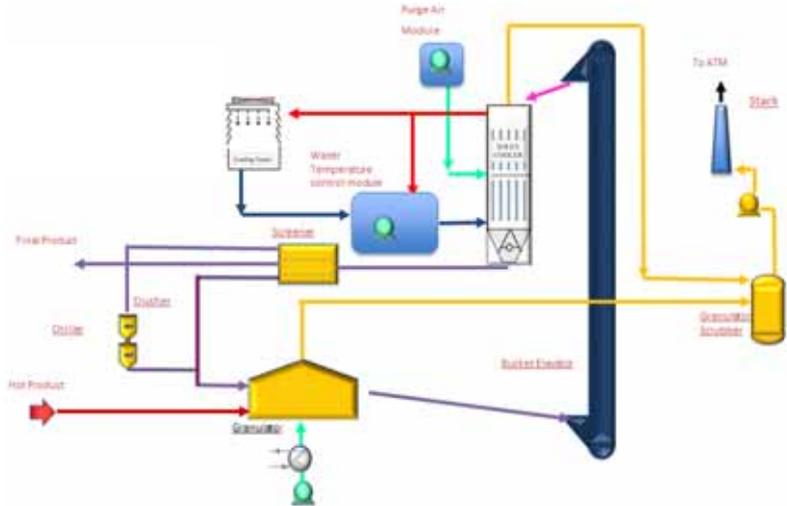


Figure 3. Fertilizer cooling is simplified and more energy-efficient using an indirect plate cooler.

There have been significant advancements in indirect plate heat exchanger technology behind the indirect plate cooler since it was first introduced to the fertilizer industry. Of these, the most major advancement was

gaining a thorough understanding of the science behind cooling a fertilizer product. Having a full understanding of this supported the design of a fertilizer cooler that guaranteed efficient cooling and long, reliable run times between scheduled cleaning in all climatic conditions.

To date, the majority of indirect plate cooler installations in fertilizer plants have been for retrofit projects and, in most cases, the primary driver was to provide catch up on the cooling capacity when the upstream plant had been debottlenecked. The technology is a logical fit in this type of project; low air emissions do not put additional loading on the air scrubbing system and a small footprint fits well into a crowded building.

Indirect plate coolers have recently been selected for the final stage of granule cooling by major process licensors in some of the latest generation fertilizer plants in the US. This has come about for two reasons. First, the technology is proven. It will perform the cooling duty and operate for long periods between scheduled cleanings. The second reason is the much lower energy consumption as a result of eliminating the high horsepower fans and air chiller needed with a fluid bed cooler.

So, how do conventional fluid bed coolers and indirect plate coolers match up? This article will take an in-depth look at and compare both units.

The conventional fertilizer cooling process

The conventional process for fertilizer cooling is to use a fluid bed cooler. Figure 1 shows a typical granulation layout. In this conventional layout, there are three main power draws: the forced draft fan, induced draft fan, and the air chiller. The fan load is straightforward and is a function of air flow rate and pressure drop across the bed. The air chiller load is more complex and will vary throughout the year, depending on ambient conditions. In many cases, the air chiller power draw will be the largest of the system.

Air chiller

A typical specified temperature of urea for storage is in the range of 40 – 45°C. Ambient air cannot be used directly to achieve this temperature. Even in temperate climates such as Alberta, Canada, where summer ambient temperatures can be 30 – 35°C, it is too warm to achieve specified discharge temperatures. In warmer and more humid climates, the problem only gets worse. This means that the air must be chilled.

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The air for the secondary fluid bed cooler needs to be chilled to approximately 10°C and then reheated to about 20°C so it is no longer saturated. The relative humidity (RH) of the air entering the fluid bed cooler must be below the critical relative humidity (CRH) of the product to avoid the product picking up moisture from the air and becoming sticky.

At 20°C, chilled air temperature is still an adequate temperature difference between the air and the required product exit temperature to achieve effective cooling efficiency. In a hot, humid climate, there is considerable heat load needed to chill the air. There is the combination of the heat load to chill the air, plus the heat load to chill the water vapour in the air and the latent heat of condensing the water out of the air. The air chiller has to be rated for the worst case conditions, otherwise it becomes the bottleneck and production needs to be cut back.

Table 1. Maximum heat load and chiller drive rating

Ambient conditions	
Average temperature	35°C
Air chiller compressor	94 RH
Enthalpy	
Ambient	124 kJ/kg
11°C, 100% RH	29.6 kJ/kg
Delta	94.4 kJ/kg
Chiller load	
Average chiller load	20 390 400 kJ/hr
Chiller power	1887 kW

Table 2. Average chiller thermal load and power draw by month

	Ambient conditions		Enthalpy			Chiller load	
	Average temperature (°C)	Air chiller compressor (RH)	Ambient (kJ/kg)	11°C, 100% RH (kJ/kg)	Enthalpy delta (kJ/kg)	Average chiller load (kJ/hr)	Chiller power (kW)
January	14	70	31.64	29.6	2.04	440 640	41
February	15	75	35.2	29.6	5.6	1 209 600	112
March	21	73	49.94	29.6	20.34	4 393 440	407
April	21	72	49.54	29.6	19.94	4 307 040	399
May	25	72	61.62	29.6	32.02	6 916 320	640
June	28	71	71.36	29.6	41.76	9 020 160	835
July	29	75	77.71	29.6	48.11	10 391 760	962
August	28	78	75.74	29.6	46.14	9 966 240	923
September	26	77	67.71	29.6	38.11	8 231 760	762
October	20	71	46.41	29.6	16.81	3 630 960	336
November	14	69	31.38	29.6	1.78	Not in use	-
December	14	74	32.65	29.6	3.05	658 800	61
Average	21.25	73.08	52.58	29.6	22.98	5 378 793	498

To give an idea, for a typical air chiller in a 3600 tpd urea plant located on the US Gulf Coast on a hot day in July, where the temperature can reach 35°C and 94% RH, the heat load would be approximately 20 million kJ/hr. It will require a chiller drive of almost 2 MW. This is a major drive for the plant and a significant capital expenditure.

Table 1 gives the calculation for the heat load and chiller drive rating for this 3600 tpd Gulf Coast example. The airflow is taken as 180 000 Nm³/hr, chilled to 10°C dewpoint and reheated to 20°C drybulb.

A second consideration is the annual operating cost. This will vary depending on ambient conditions and local power cost. Using the above example, on an average day in January, the ambient temperature is 14°C at an RH of 70%. In these conditions, the chiller is barely ticking over, drawing only about 40 kW for the thermal load, compared to over 1 MW on an average day in July, when the temperatures are approximately 29°C and RH of 75%. Looking over 12 months, the average chiller load is about 500 kW, and at a power cost of 10 c/kWh, and 8000 hr/yr, this is an annual electrical cost of US\$400 000.

Table 2 gives the average chiller thermal load and power draw by month.

Fans

In a fluid bed cooler system, there are typically fans upstream (forced draft) and downstream (induced draft) of the fluid bed. Estimated power consumption is 180 kW for the forced draft fan and 360 kW for the induced draft fan. An overall summary of the estimated power draw for a fluid bed cooler is detailed in Table 3.

Progress in fertilizer cooling

As mentioned previously, the alternative to the fluid bed cooler is the indirect plate cooler system. Incorporating an indirect plate cooler system eliminates the need for a

chiller unit and scrubber fans. The typical process flow of the secondary cooling in a urea granulation plant using an indirect plate cooler is shown in Figure 3. The power draws for this system are the water pump on the water module and the bucket elevator, and these draws are substantially lower than a fluid bed cooler, as shown in Table 4.

Making a comparison of the fluid bed cooler configuration in Figure 1 and the newer indirect plate cooler system configuration in Figure 3 in terms of energy consumption, it can be observed that there are substantial energy savings

Table 3. Estimated power draw for a fluid bed cooler

	Consumption in kW
Forced draft fan	180
Induced draft fan	360
Air chiller compressor	500

Table 4. Power draws for the indirect plate cooler process

	Consumption (kW)
Purge air blower	45
Waste pump	20
Extra bucket elevator	20

Table 5. Total energy costs – fluid bed cooler versus indirect plate cooler

	Fluid bed cooler (kW)	Indirect plate cooler (kW)
Air chiller	498	-
Air blower	180	45
Fans ID	360	-
Cooling water pump	-	20
Bucket elevator	-	20
Total power consumption	1.038	85
Total energy savings per hour	953	

with the new configuration. Table 5 shows a comparison of the total energy costs.

The verdict

Indirect plate coolers have proven to be reliable and efficient for cooling fertilizers. There are now over 100 in service across the world in every type of fertilizer processing, including urea, ammonium nitrate, NPKs and phosphates. Some of these units have been in service for more than 25 years, and developments over the last few years have addressed some of the early concerns with caking. These concerns were tackled by carefully controlling the water temperature and by adding low volume dry purge air to achieve reliable operation and long run times between cleaning.

Reduced energy costs have been the primary focus of this article and the example suggests that there is significant energy, and thus operating cost, savings per year with the indirect plate cooler. A large component of the cost savings is the elimination of the air chiller. In addition to operating cost savings, three large energy drains of 180kW, 360kW, and 2000 kW are eliminated and replaced by two small pieces of equipment that total less than 50 kW in energy usage. This serves as an example of forward progress within a long-standing industry. **WF**

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Minimising downtime and maximising plant efficiencies remain among the key priorities across the chemicals and process industries, which rely on the innovation of technology-led companies from within its supply chain to meet essential targets.



Figure 1. A Flexitallic Change gasket.



Figure 2. Typical heat exchanger equipment.

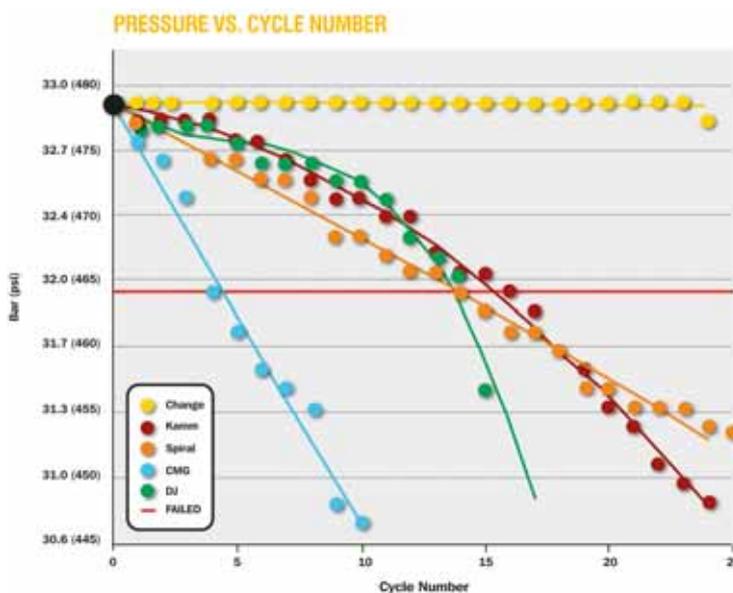


Figure 3. Pressure versus cycle number results.

Flexitallic has been delivering gasket and sealing solutions to the industry for over a century, focusing on the particular challenges faced by sectors, such as the ammonium nitrate and nitric acid production sector, with the aim of improving the integrity of bolted joints for piping systems and process equipment in order to achieve leak-free operations.

The cyclical nature of the temperature ranges handled by heat exchangers employed in the production process has, in the past, led to leakages becoming a chronic issue and a challenge to plant engineers.

Although specific process designs differ, typically downstream of the burner vessel are a series of heat exchangers that are subject to high temperatures and an oxidising service. Whilst the burner gaskets are changed when the catalyst changeover is required, the nitric acid heat exchanger train gaskets are not changed. Instead they are exposed to the shutdown and restart thermal cycle, which adds an additional strain on the bolted joint especially the gasket.

The multiple cycles of extreme temperatures and pressures in heat exchangers can lead to unplanned maintenance and downtime due to the need to replace any gaskets that are unable to tolerate these tough conditions.

Previously, the sealing options available have been unable to operate across a broad temperature spectrum, which has led to leak mitigation rather than eradication. However, even a single gasket failure can lead to costly plant shutdowns and lost production time, while gasket replacement during inspection and maintenance programmes can extend the planned shutdown, thereby impacting plant operations.

Flexitallic's Change™ gasket offers a major benefit to the fertilizer industry as it addresses these issues through its specialist design. It is capable of operating at high temperatures in an oxidising environment, and can also cope with repeated extreme thermal cycles.

The Change gasket is particularly effective when used within heat exchangers, which makes it suitable for the extreme process demands used in the manufacturing of nitrate acid and the production of syngas. High temperatures alongside a highly oxidising process media and extreme thermal cycling are the most challenging environments for the fluid sealing industry.

Processes at fertilizer plants involving heat exchangers can be particularly demanding and can involve elevated temperatures, high pressures, and cyclic operation, especially in heat trains and burner applications. This is compounded by the fact that equipment is often old. Additional requirements for improved asset efficiency and emissions compliance place ever-increasing demands on gasket performance.

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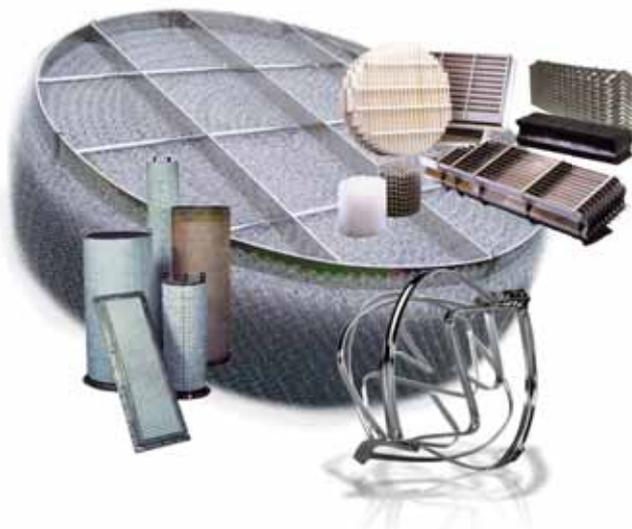
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Operators of fertilizer plants are constantly working to improve the profitability of their production assets. The ability to expand the capacity of an operation by selecting the vessel internals best suited for a specific application can result in a cost effective solution as well as a shorter turnaround.

While capacity is of primary interest, Koch-Glitsch can also help operators address concerns with efficiency, product purity, operation reliability, and the environmental impact of processing operations.

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To address such issues, extensive research and development work was undertaken that resulted in the production of the Change gasket. It has been designed to provide the optimum combination of both stiffness and resilience, so is capable of creating a high performance seal under the conditions of a long-term cyclic service.

Change offers substantial benefits over traditional gasket styles that are more commonly used in heat exchangers, including metal jacket gaskets, corrugated metal gaskets, spiral-wound gaskets and kammprofile gaskets. It also offers long-term sealing performance, particularly in bolted connections that are subject to thermal transients.

The manufacturing approach to Change involved producing a gasket from a wire strip, in order to offer both maximum manufacturing flexibility and cost savings alongside optimum material usage. The strip is spirally wound; however, the strip profile and thickness differs significantly from that used in a standard spiral-wound gasket, being five times thicker.

Upon winding, the unique profile and increased thickness of the wire results in a stiff yet resilient interlocked structure with improved handling characteristics compared to that of standard spiral-wound gaskets.

The gasket's capabilities have been proven through a series of studies, including radial shear testing (RAST), shell extended thermal cycling, leakage and compression testing, which all yielded positive results.

For example, across a 24-cycle 'pressure versus thermal cycle' test at 302°C (replicating industry conditions), a bolted joint using Change lost just 1.5 psi, outperforming other gasket tested by at least nine thermal cycles.

Compression tests revealed that, based on the high level of stored energy, Change recovers almost five times better than kammprofile and double jacketed gaskets. RAST (originally developed by PVRC/ASME along with Ecole Polytechnique at the University of Montreal), was undertaken to simulate the differential expansion and contraction of heat exchanger flanges. The test was conducted over 100 thermal cycles with nitrogen pressurising to 40 bar and leak test every 20 cycles.

The flange was a tongue and groove type, as is typical of a heat exchanger, and the gasket size was 453 x 427 mm (nominal 3.2 mm thick). The lower flange was cycled to 300°C and the upper flange was water cooled, thus maximising differential growth in the system and generating approximately 0.8 mm radial shear on the gasket. The key measurement however, was the bolt relaxation as this is considered a measure of the resistance of the gasket to radial shear.

The gasket survived the test with negligible visible damage, and showed only a 15% relaxation in bolt stress after 100 cycles. By comparison, the next best performing was the spiral-wound gasket with a bolt relaxation of approximately 25%. Flexitallic's gasket exhibited no major leakage in the test, which was in stark contrast to the jacketed type gasket.

Change has also consistently performed in tough environments. For example, CVR Energy recently installed the gasket in one of its nitric acid plants. The initial installation was in a steam superheater heat exchanger with NO_x operating at 125 psi and 965°F. The previous gasket was a double jacketed gasket. The unit was located between an air heater and primary waste heat boiler.

After three to four thermal cycles, both the upstream and downstream process flanges would develop leaks and gaskets were replaced at every gauze change. In October 2013, Change gaskets were installed in both process flanges. The original Change gaskets remained in service for several leak-free years. The gaskets were replaced in 2016 due to a tube leak in the superheater and also remain in service leak-free after numerous thermal cycles.

There are several other reported successes from fertilizer facilities using the Change gasket in various applications, including process piping, natural gas preheater exchangers, ammonia burners and tail gas heat exchangers. The gasket is also being used in a range of applications in other industries, such as the refining, power and petrochemical sectors.

This gasket can create a tight seal that responds to temperature cycles without the risk of overtightening or loosening of bolted joints to compensate. This efficacy can be enhanced by the use of filler materials, which are incorporated during the gasket winding process.

Once wound, both gasket sealing faces are covered with a layer of soft facing material, providing low-stress sealing and good performance on poorly-finished flange surface faces. For chemical processes, materials such as PTFE can be applied or, in the case of fertilizer production, Flexitallic's high-temperature material, Thermiculite®, is often used.

Based on naturally-occurring vermiculite, Thermiculite materials are able to operate at both ends of the temperature spectrum, including up to temperatures in excess of 1000°C. This makes it suitable for nitric acid production. Nitric acid heat train exchangers undergo regular heat cycles as the reactor catalyst is replaced every 70 – 90 days. However, during this very exothermic process, gaskets are often not replaced, and with temperatures reaching up to 871°C, sealing products can be put under extreme pressure and can become prone to failure.

However, because vermiculite is an inorganic silicate, Thermiculite possesses a strong resistance to very high temperatures and aggressive chemicals, and has passed the stringent API 6FB fire test.

Thermiculite and Change can be used as a single solution, replacing the need for a range of materials – none of which have provided a lasting solution at elevated and cycling temperatures. Real savings can be made in terms of reduced maintenance costs, reduced downtime, rationalisation of inventory, emissions compliance and improved safety through vastly-improved joint integrity. **WF**

THE RED CARPET TREATMENT

Dr Gene Shelp and Dr John Motto, ENPAR Technologies Inc., Canada, look at effective ammonia treatment for commercial, industrial and municipal applications.

Effective ammonia treatment systems are becoming more important in an increasingly environmentally conscious world. ENPAR's AmmEL systems represent cost-effective and environmentally sound treatment processes for the removal of dissolved ammonia from industrial and municipal water, including wastewater, storm water, sewage effluent and mine wastewater.

At the core of the system is an electrochemical reactor that converts the total ammonia directly into gaseous nitrogen without producing carcinogenic nitrate or the greenhouse gas

THE AmmEL-LC PROCESS

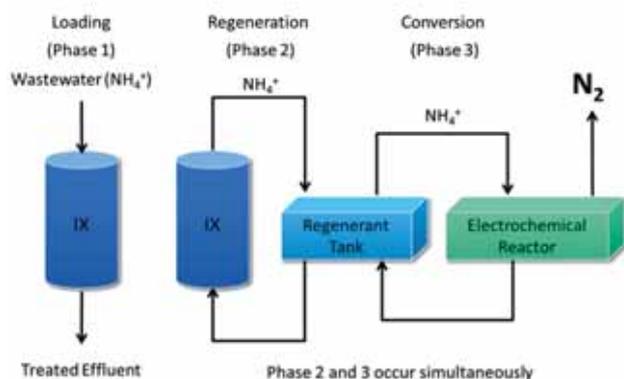


Figure 1. The AmmEL-LC process.

Table 1. Difference between AmmEL-LC and AmmEL-HC specifications

Specification	AmmEL-LC	AmmEL-HC
Operational $\text{NH}_3\text{-N}$ concentration	< 200 mg/l	> 200-1000s mg/l
Ammonia removal method	Ion exchange	Strip and scrub
Ammonia recovery method	N_2 (destructive via electrochemical reactor)	N_2 (destructive), or $(\text{NH}_4)_2\text{SO}_4$ (can be recovered and re-used)

nitrous oxide. Responsibly managing ammonia will reduce the toxicity, inhibit the proliferation of algae and improve the overall water quality of receiving water bodies.

In contrast to conventional biological processes, low temperatures do not negatively affect the efficacy of ENPAR's electrochemically based systems. This enables the management of ammonia in northern regions of the globe even under extremely cold conditions (0°C).

The system takes the 'N⁴O Approach' to ammonia treatment:

- No nitrate: as it is a carcinogen.
- No nitrous oxide: as it is a known greenhouse gas.
- No reduction in water quality.
- No algae.
- Only environmentally-friendly nitrogen gas.

Some of the advantages of the AmmEL technology compared to conventional systems include:

- It is capable of achieving very low concentrations of ammonium in the treated water (<1 mg/l).
- Neither nitrate nor nitrous oxide is produced (releasing 1 kg of N_2O into the atmosphere is reported as being equivalent to 298 kg of CO_2),¹ so the system should qualify for greenhouse credits.
- The efficacy is independent of temperature.
- Water with high hardness, high-suspended solids and limited organic compounds can be treated.
- The system has a relatively small footprint with rapid startup and shutdown, and is fully automated and

remotely monitored, therefore requiring little maintenance.

- The system incurs a lower capital cost than current conventional systems of similar size and capabilities.
- There is no bio-fouling of columns and, therefore, no reduction in ammonium adsorption efficiency.

There are two versions of the AmmEL system: AmmEL-LC and AmmEL-HC. Table 1 gives the specifications for each version.

AmmEL-LC

AmmEL-LC is a two-stage continuous process that incorporates high efficiency ion exchange columns that are linked to an electrochemical reactor. The process involves pumping wastewater-containing ammonia into sequential ion exchange columns that contain a naturally occurring ion exchange material. Loaded columns are regenerated using a simple salt solution. The sequence of loading and regeneration changes automatically to provide maximum treatment.

ENPAR's research team developed a modified natural ion exchange material capable of maintaining high ammonium removal rates while precluding the absorption of calcium, magnesium and strontium ions. These ions, when transferred to the regenerant solution of the AmmEL-LC system, had been targeted as a source of fouling of the electrochemical reactor. The modified ion exchange material allows the calcium, magnesium and strontium to be carried through with the treated effluent while maintaining a high rate of ammonium removal, precluding the transfer of hardness to the regenerant solution.

Municipal wastewater treatment plants can employ AmmEL-LC to treat clarified secondary effluent, and it can function as a complementary system for treatment facilities during cold weather conditions when effluent temperatures approach 0°C and nutrient removal in biological reactors slows down. With a high removal rate immediately upon startup and the ability to maintain high ammonia removal efficiency when treating waste streams at 0°C , the system can be applied as a polishing step to remove ammonia during the final treatment stage prior to discharge.

Treating contaminated groundwater

ENPAR was contracted by an engineering firm to design, manufacture and deliver an AmmEL-LC ammonia treatment system to a project site in the Middle Atlantic region of the US. The source of the ammonia-N ($\text{NH}_3\text{-N}$) was minor releases of diesel exhaust fluid (DEF) due to an end-user error (spillage) during the filling of DEF tanks on diesel vehicles. During periods of rain or snowmelt, or when the ground near the filling stations was washed, the DEF was washed away and collected in a retention pond near the site. Over time, the DEF was converted to ammonia by bacteria present in the pond water.

Concentrations of $\text{NH}_3\text{-N}$ in the collection pond derived from the urea in the DEF resulted in exceedances of the site's permitted ammonia discharge limit. In order to meet the discharge requirements, ENPAR delivered a

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mobile AmmEL-LC unit, which was constructed in a 45 ft shipping container. The unit was designed to reduce concentrations of $\text{NH}_3\text{-N}$ from 200 mg/l to less than 1.5 mg/l. The unit operates at a maximum system flowrate of 5 gal./min. (18.9 l/min.) under the most adverse treatment conditions created by the range of water chemistry parameter values encountered at the site.

Following commissioning, the automated AmmEL plant met the specified design criteria resulting in ammonia concentrations of less than 0.2 mg/l $\text{NH}_3\text{-N}$ in the treated water. This system was designed to operate continuously or intermittently during periods of limited precipitation.

Ammonia removal from storm water run-off

ENPAR's AmmEL-LC system was also used in a pilot study at a municipal bio-solids storage facility located in Southern Ontario, Canada. The objective of the pilot-scale tests was to demonstrate that the system could reduce the concentration of ammonia in the storm water pond to below 3.0 mg/l $\text{NH}_3\text{-N}$. These guidelines were required by a Certificate of Approval issued to the region by the Ontario Ministry of the Environment regarding the direct discharge of water during the periods 1 March – 31 May and 1 November – 15 December.

The region had recently been granted approval by the Ministry of the Environment to expand the existing storm water management pond. ENPAR needed to demonstrate that the AmmEL system could treat the additional storm water on-site at a lower cost when compared to the alternative of hauling storm water to a municipal sewage treatment plant.

Initial testing was performed with the system operating for 6 hr a day for three weeks. During that period, samples of inlet and treated water were taken twice during each 6 hr run. A total of 72 samples (36 inlet samples and 36 outlet samples) were analysed for $\text{NH}_3\text{-N}$ concentration by ion selective electrode within 24 hr of arriving at ENPAR's facility.

The $\text{NH}_3\text{-N}$ concentration of the inlet storm water at the beginning of the testing period was >20 mg/l and decreased gradually to approximately 14 mg/l by the end of the test period. Treatment of the storm water using

AmmEL reduced the concentration of $\text{NH}_3\text{-N}$ to <2.6 mg/l with an average value of 0.55 $\text{NH}_3\text{-N}$ mg/l. Assuming an average value of 17 mg/l for the inlet stream, this represented a 96% reduction in the total concentration of $\text{NH}_3\text{-N}$ in the storm water after treatment.

Following the initial testing period, the system was operated on a 24 hr basis for one week. The inlet $\text{NH}_3\text{-N}$ concentration was 2.25 mg/l due to extended periods of rain and dilution in the retention pond. However, following treatment, an average $\text{NH}_3\text{-N}$ concentration of 0.35 mg/l was measured in the outlet samples. This corresponded to an average of 84% removal of the total $\text{NH}_3\text{-N}$ in the storm water samples after treatment.

This pilot-scale test of the AmmEL system demonstrated that the technology is both a viable and practical method for removing ammonia from contaminated water. It has the additional advantage of converting ammonia directly to N_2 gas. As nitrate is not produced in the process, it is possible to reduce the total nitrogen loading and not just the ammonia loading. This is extremely valuable, given that regulators are now contemplating the implementation of discharge regulations based on total nitrogen ($\text{NO}_3\text{-N}$ and $\text{NH}_3\text{-N}$) loading.

AmmEL-HC

AmmEL-HC is a two-stage ammonia removal process consisting of strip and scrub columns that are linked to an electrochemical reactor. At the core is an electrochemical reactor, which converts ammonia directly to gaseous nitrogen without producing nitrates or greenhouse gases. This technology is suited for treatment of municipal wastewater and storm water, as well as industrial process water and contaminated groundwater.

Municipal wastewater treatment plants can employ AmmEL-HC after sludge dewatering processes in order to prevent ammonia toxicity in the activated sludge process. Like its counterpart, the AmmEL-HC can function as a complementary system for treatment facilities during cool weather conditions when nutrient removal in biological reactors slows down. With a high removal rate immediately upon startup, it is able to divert excess loading from existing activated sludge processes during acclimation periods or system upsets.

The strip and scrub approach comprises three phases:

- Stripping ammonia gas from the wastewater.
- Scrubbing the ammonia back into a liquid stream.
- Electrochemically converting the ammonia to free nitrogen gas.

The pH of the wastewater is increased before being pumped into the top of the stripping column. Air that is pumped into the bottom of the column transfers the ammonia from the wastewater to the liquid in the scrubbing column. The scrubbing liquid is then transferred to the electrochemical reactor where the ammonia is converted to nitrogen gas.

An alternative version of the AmmEL-HC, the AmmEL-Rx has application for treating high ionic

THE AmmEL-HC PROCESS

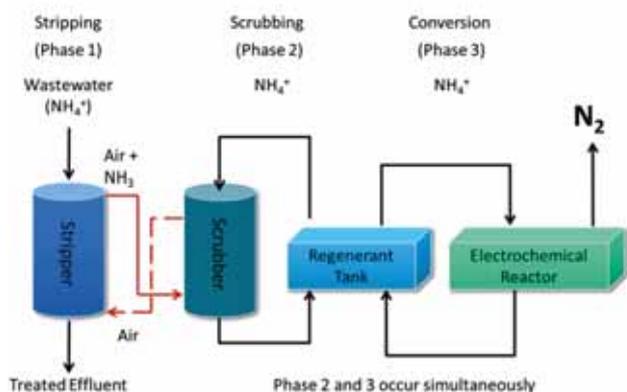


Figure 2. The AmmEL-HC process.



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Table 2. AmmEL-HC optimisation experiments for centrate at 24 °C

Run code	Unit	416	414	415	417
Centrate flow rate	l/min.	3	4	4	5
pH of stripper inlet		10.1	10.1	10.1	10.1
pH of stripper outlet		8.2	8.2	9.6	9.5
Reactor current	A	500	600	500	600
Inlet NH ₃ -N	mg/l	903	960	823	906
Outlet NH ₃ -N	mg/l	69	234	215	247
NH ₃ -N removal	%	92	76	74	73
Mass NH ₃ -N removed	kg/m ³ centrate	0.83	0.73	0.61	0.66
Reactor power	kWh/m ³ centrate	16.9	15.8	12.7	12.8
Power other	kWh/m ³ centrate	14.3	10.7	10.7	8.6
Power cost at US\$0.09/kWh	US\$/m ³ centrate	2.81	2.39	2.11	1.93
HCl consumption	l/m ³ centrate	3.52	3.68	1.35	1.33
HCl cost at US\$0.20/l	US\$/m ³ centrate	0.7	0.74	0.27	0.27
NaOH consumption	l/m ³ centrate	3.99	3.85	3.96	4.11
NaOH cost at US\$0.50/l	US\$/m ³ centrate	2.00	1.93	1.98	2.06
Total operating cost	US\$/m ³ centrate	US\$5.51	US\$5.05	US\$4.36	US\$4.25
Total operating cost	US\$/kg NH ₃ -N removed	US\$6.61	US\$6.96	US\$7.17	US\$6.45

strength waste streams containing a high chloride concentration (e.g. >5000 mg/l chloride). In this case, the ammonia in the wastewater stream can be treated directly by the electrochemical reactor, bypassing the need for an initial strip and scrub stage.

Ammonia removal from municipal centrate waste streams

The removal of high concentrations of ammonium (>800 mg/l) from a municipal centrate waste stream was successfully demonstrated using ENPAR's AmmEL-HC small scale commercial system at the Wastewater Research Training Centre, operated by the Edmonton Waste Management Centre of Excellence (EWMCE) in Edmonton, Alberta, Canada.

The AmmEL system consisted of countercurrent air-stripping and air-scrubbing packed columns operated in a closed-loop, alongside an electro-chemical reactor. The stripper column was 40.6 cm in diameter and 5.8 m in height, while the scrubber column was 40.6 cm in diameter and 4.3 m in height. The stripping column and scrubbing tower transferred the ammonia from the waste stream to a brine solution. The brine solution contained the electrolyte of the closed-loop electrochemical circuit, which was pumped through the electrochemical reactor where the ammonia was then oxidised to nitrogen gas. The nitrogen gas was subsequently released to the atmosphere.

The electrochemical reactor was operated at a current between 500 and 600 A, and at a potential of 6.2 V. The pH of the centrate was adjusted to a value between 8 and 10 (depending on centrate temperature),

immediately before it entered the stripper column. The pilot-scale unit was operated at a liquid flowrate of 3 – 5 l/min., and a gas flowrate of 12 m³/min.

Within the range of conditions tested by the EWMCE technical team, the system proved capable of removing up to 90% of the ammonia in the waste stream and converting virtually all of the captured ammonia to nitrogen gas. The ammonia concentration measured in the reactor exhaust air stream was below the 5 mg NH₃-N/l detection limit. The cost of treatment (including the power and chemicals) was approximately US\$4.25/m³ – US\$5.51 of centrate containing 825 – 960 mg NH₃-N/l and at 24 °C (Table 2).

The operating costs were reduced significantly when using higher centrate temperature. The higher temperature AmmEL-HC feed water translated to a reduced NaOH consumption, as ammonia solubility in water is decreased at higher temperatures. Therefore, a smaller pH adjustment was necessary.

The AmmEL technology is a disruptive technology that efficiently and effectively converts ammonia to nitrogen gas. Two versions of the AmmEL are available depending on the ammonia concentration of the wastewater. The AmmEL-LC is an effective approach for wastewater with ammonia-nitrogen concentrations below 200 mg/l, producing treated effluent containing less than 1 mg/l NH₃-N. The AmmEL-HC is best suited for treating wastewater with NH₃-N concentration above 200 mg/l. Both technologies convert ammonia to nitrogen gas whilst bypassing formation of the carcinogen nitrate or the greenhouse gas nitrous oxide and effectively reducing total nitrogen loading in the environment.

The AmmEL system is a viable ammonia solution to the fertilizer industry, for the treatment of groundwater, storm water and air scrubber condensate water containing elevated levels of ammonia. The AmmEL-LC system can be utilised in a continuous operation pump-and-treat programme to treat groundwater that contains ammonia. For the treatment of ammonia-containing storm run-off water, the AmmEL-LC can be used as a tertiary process or used in a batch treatment mode. The AmmEL-HC can be utilised to treat air scrubber condensate, which may contain higher levels of ammonia. When used in these applications, both the AmmEL-LC and AmmEL-HC convert ammonia to environmentally-friendly nitrogen gas that can be safely released to the atmosphere. **WF**

Reference

1. 'CO₂ equivalents', *Climate Change Connection*, <https://climatechangeconnection.org/emissions/co2-equivalents/>

FROM RECOVERY TO REMELTING

MARK GILBREATH,
MATRIX PDM ENGINEERING,
USA, OUTLINES INFRASTRUCTURE
CONSIDERATIONS FOR GLOBAL DRY
SULFUR LOGISTICS.



Sulfuric acid – the most commonly used chemical in the world – is a primary feedstock in the production of fertilizers, as well as in the manufacturing processes of many other products.

Today, more and more sulfur is being generated as a result of increased global oil and gas production and environmental regulations that demand lower sulfur

content in transportation fuels. The result is an evolving need for efficient sulfur storage and transportation logistics, which has, in turn, led to increased reliance on the production and transportation of dry formed sulfur that can be carried in bulk collier ships. Concurrently, large new facilities that have the capacity to form over 1000 tpd of sulfur



Figure 1. Wet prilled sulfur being conveyed from a sulfur prilling unit to a pile for open storage.



Figure 2. Material handling system including marine structures and shiploaders designed to handle either molten or prilled sulfur.



Figure 3. A 1 million tpy sulfur remelter with lime silo, external heat exchangers and a scrubber.

are being constructed in Canada, India, Iran, Kuwait, Malaysia, Mexico, Turkey and Vietnam.

From recovery of molten sulfur, to dry bulk solid, and back to molten sulfur, this process requires specialised technology and infrastructure to ensure safe handling, transportation and storage. This article focuses on those processes and infrastructure.

Degassing units

Sulfur that has been degassed to lower hydrogen sulfide (H_2S) results in a higher quality and much safer formed dry sulfur prill. Claus liquid sulfur contains 250 – 350 ppmw of dissolved H_2S/H_2S_x . With a long logistic supply chain, dangerous H_2S is released during storage, handling and transportation. This is particularly dangerous due to potential H_2S buildup in a ship's hold during ocean transportation.

Key drivers for sulfur degassing are safety, health and environmental improvements. Degassing units that include contactor, cooler and mechanical systems are readily available in vertical modular packages.

Sulfur forming units

Products that can be handled as dry bulk solids are formed by either dry pastillation, granulation, or wet prilling. The objective is to solidify molten sulfur into a product with a shape and integrity that will create the minimum amount of fine sulfur particles during transport, transfer, storage and reclamation. Sulfur dust is both a health hazard and presents potential for explosion if collected in a confined space with an ignition source.

Pastillation units spray water under a steel belt on which sulfur drops are deposited to produce pastilles. Meanwhile, granulation units use water sprayed into a rotating drum for cooling the liquid sulfur that is sprayed onto a seed curtain to form granules.

Prilling introduces liquid sulfur at the top of a forming tank for direct countercurrent heat exchange with water to produce prills that are withdrawn from the bottom of the forming tank via screens. Unlike a pastillation or granulation unit, prilling units are readily adaptable for modular construction. Major components include the forming tray, fume hood and scrubber (if required), forming tank, dewatering screen and dry product conveyors. Mechanical equipment includes the process water and cooling tower supply pumps and cooling tower with fan units.

This wet formed prill also benefits from modern process improvements for moisture control (2%), size uniformity, high capacity and ability to meet international commercial specifications. Apart from in very cold climates, process building enclosures are not required.

Dry sulfur material handling

A major advantage of dry sulfur in the global logistics chain is the ability to store, handle and transport it as a dry bulk product. Depending on facility owner preferences, it is acceptable to store dry sulfur in open storage stock yards. However, these yards are subject to windborne contamination (such as sand) and increases in free moisture due to rain. While dry storage in buildings will protect the sulfur from the elements, it also requires greater dust management.

Wet prilled sulfur (Figure 1) is less dusty from the time it is produced. There are also a number of commercially-available dust suppression products that work effectively if properly distributed at transfer

points along the material handling conveyor system.

Control of acidity is an important consideration for protecting material handling equipment and facilities. While total stainless steel construction would be ideal, it tends to be more expensive than most facilities can justify. Experienced providers of sulfur material handling systems understand this and will generally be able to offer a more cost-effective solution. That said, regardless of whether open or dry building storage is used, sulfur acidity will biologically increase over time. The use of additives before a material is placed in ship holds or at ports of storage for periods of over a few weeks can slow this activity.

Shiploading and unloading

Being able to load ships with dry sulfur at the origin or unload ships at the consumption destination is greatly enhanced if existing dock facilities are available (Figure 2). The capital expense for basic new dock construction in the US starts at over US\$30 million, excluding the cost of material handling conveyor systems, stockpiles and shiploading/unloading equipment.

Co-location of sulfur handling ship facilities at an existing dock can lower capital construction costs. However, berth occupancy minimum times and potential per diem ship costs for a shared dock must also be considered.

Large heavy longitudinal travelling shiploaders/unloaders provide the greatest flexibility for working vessels at rates of over 3000 tpd. These machines also have the highest installed capital cost, requiring substantial existing and new dock structure to support. In many cases, smaller fixed-arm shiploading equipment will have the lowest capital cost, but will also mean lower unloading rates (approximately 1000 tpd) and, unless one hold is being loaded for small cargo, the ship must be repositioned during loading/unloading.

Ships can be loaded/unloaded with dry sulfur using basic cranes and ships gear. While these simple systems have low capital costs, they require more manpower and have higher operating costs over the life of the facility.

Land transportation

If the sulfuric acid plant and associated melter is a distance from the ship dock, dry sulfur can be transported by rail or truck. The capital and operating costs of rail or truck loading/unloading facilities need to be taken into account accordingly. Often, only the cost of transportation is included in a business model, with truck and rail facilities easily overlooked or underestimated.

Sulfur remelting facility

A sulfur remelter is required to transform dry sulfur to molten sulfur, to then feed the sulfuric acid plant. Single melter vessel systems using external heat exchangers (Figure 3) can be provided with a capacity of well over 4500 tpd, which adds heat to the melter system through



Figure 4. Three modular pressure leaf filter units at a 1 million tpy sulfur remelting facility.

the use of external heat exchangers, without the use of internal coils in the melter vessel. External heat exchangers are much more efficient to maintain and operate than systems that include internal coils.

Dry sulfur is added to the melter vessel at a controlled rate. Heated molten sulfur is also added at the top of the melter. Properly designed mixing and the creation of a controlled vortex facilitate the highly efficient melting of the dry sulfur. Molten sulfur then flows from the melter vessel into an elongated pump tank. The pump tank and associated vertical shaft pumps circulate the molten sulfur through the heat exchanger/melter vessel system. Importantly, the pump tank also allows for non-meltable contaminants and any foam to settle out.

Filtering and molten sulfur storage

The last step before molten sulfur can be stored is post-melting filtering. This is typically completed using pressure leaf filter units at the remelter facility (Figure 4). These units use filter leaves that are pre-coated with filter media, such as diatomaceous earth. As filter efficiency decreases, the filters are taken offline, opened and the filter cake removed.

These properly sized and designed leaf filter units are readily adaptable to modular fabrication away from jobsite and transported as complete units by truck, barge or ship. Polishing cartridge filters can also be provided depending on the acid plant ash specification in cases where adequate filter screens and pre-coat is not available.

Molten sulfur is then pumped from the filter system to the molten sulfur storage tanks that provides feed to the sulfuric acid plant. Molten sulfur storage tanks require proper design and construction in order to maintain uniform heat and temperature control, as well as proper and safe venting.

Conclusion

Taking note of the necessary considerations and requirements for the production of dry sulfur, global ocean transportation and remelting is an important factor as decisions are made regarding changes in markets, new facility locations and type. **WF**

FINISHING TOUCHES

Wilfried Dirx and Barbara Cucchiella, Stamicarbon, the Netherlands, explore some of the latest developments in fertilizer granulation technologies.

After initial development activities were conducted in the 1970s, the first commercial test facility was contracted in 1998 at the Grodno Azot plant, Belarus, in which a small granulation unit was completely converted to Stamicarbon's granulation process. After this successful introduction, scaling up took place at the Agrium urea complex in Fort Saskatchewan, Canada. Here, two existing granulation lines of 625 tpd were converted to the Stamicarbon granulation process. The plant commenced granulation production in 2003 and is still performing to the satisfaction of the customer today.

A flow diagram of the original Stamicarbon granulation process is shown in Figure 1. Key to the success of the process are the film spraying nozzles (Figure 2).^{1,2} Due to the characteristics of the film spraying nozzle, as well as the fact that the Stamicarbon granulation process applies a 98.5% melt concentration, the process results in a high-quality end product because the granules are built up layer by layer. Before a new layer of liquid melt is applied on the seed particle, the layer on the particle is completely solidified.

Compared to other fluidised bed granulation processes, considerable OPEX savings can be achieved due to a reduced formaldehyde content in the final product and the low dust formation, leading to less interruptions for cleaning.

Simplified design

To save on both investment and operating costs, Stamicarbon omitted two fluid bed coolers and their related equipment, introducing a simpler concept in 2008. The fluid bed granulator cooler was dispensed by increasing the length of the cooling zone in the original granulator.² The fluid bed product cooler was replaced by a solids flow cooler. The respective granulator cooler scrubber with all necessary pumps and fan were omitted.

The liquid melt is fed to the granulator at a temperature of 140°C and in a concentration of 98.5% urea plus biuret. In the granulator, bed temperatures are maintained at 102 – 110°C. However, at the last stage, the end product is cooled further (85°C) compared to the original design.

After passing the lump screen, the product is lifted using a bucket elevator to the classification equipment. The complete solid product flows through the main screens. The coarse product is fed to the crusher after cooling to 70°C. The crushed product and the fine recycle flow are combined and recycled to

the granulator as so-called seeds. The on-spec product in the outlet of the main screens is cooled to storage temperature in a solid flow cooler, which makes use of cooling water and no fluidisation air.

The dust loaded air from the granulator and all the de-dusting points is collected and fed to a single granulator scrubber. The reduced number of equipment items compared to the original process flow diagram resulted in a reasonable reduction of overall plant costs.

Total CAPEX cost reductions are not only achieved by taking out equipment, but there are also savings in transportation (shipping) cost, cost of insurance, effect of reduced footprint, construction, etc. Having less equipment will also result in a reduction in maintenance costs and OPEX savings.

To reduce the amount of fluidisation cooling air on hot working days, a water injection system is provided in the discharge of the fluidisation air fan. This is only operated on hot days to increase relative humidity and reduce air consumption.

The Shahjalal experience

In 2011, Stamicarbon signed a 1760 million tpd melt and granulation licensing contract for the Shahjalal plant (located close to Sylhet, Bangladesh), where its simplified granulation design was implemented. Startup and commissioning of the plant successfully took place in 2015 (Figure 4).

With a minimal amount of equipment, this plant demonstrated an investment saving of at least 20%. It started up easily, showing reliable and smooth operation with minimal operator attention required. The plant's power consumption meets expectations and is 20% lower than the conventional design. Maintenance costs were reduced by the elimination of three fans and other equipment.

To reduce the CAPEX of the granulation plant further, Stamicarbon is working to reduce the total height of the building by relocating strategic equipment to lower elevations.

Scrubber technology

To meet the more stringent particulate matter emission regulations, EnviroCare and Stamicarbon have developed a multi-stage process for effective and efficient scrubbing of urea particulate matter and ammonia from granulation plants. With this technology, emissions of less than 10 mg/Nm³ for dust and ammonia can be achieved.^{3,4}



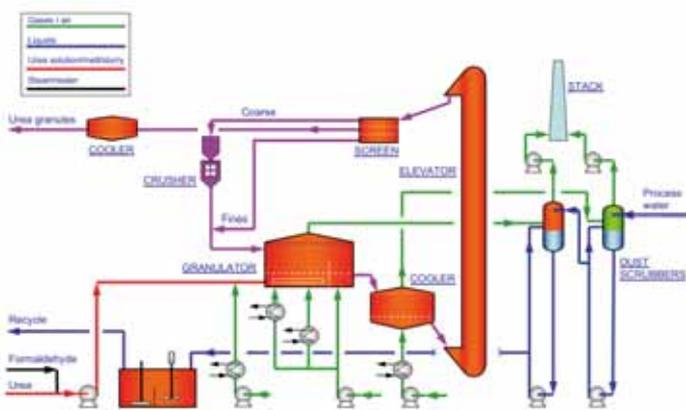


Figure 1. A process flow diagram of Stamicarbon's original granulation process.



Figure 2. Stamicarbon film spraying nozzles.

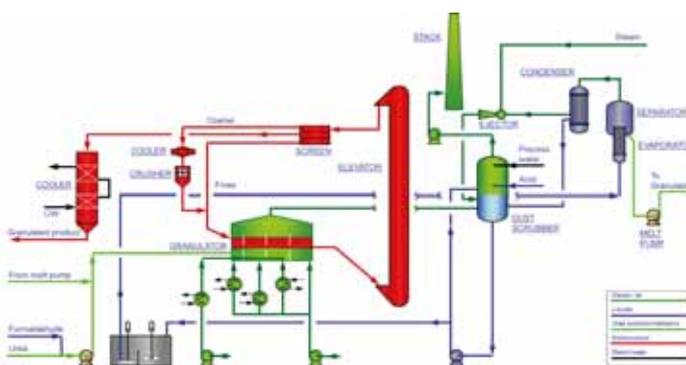


Figure 3. A process flow diagram of Stamicarbon's simplified granulation process.

During the granulation process, sub-micron dust is generated, which is largely responsible for higher emission values. While scrubbers of older technologies easily scrub larger particles, the presence of a high degree of sub-micron dust requires a new capture approach. Moreover, to efficiently remove ammonia, an acid solution needs to be injected.

The EnviroCare MicroMist™ Venturi (MMV) scrubber (Figure 5) contains six stages that progressively treat and clean the off-gas.⁵

In stage one, the exhaust gas is cooled, saturated and most of the coarse particulate urea dust is collected from the gas stream.

The concentrated urea solution is typically 35 – 45% urea (by weight).

Downstream of the first quench zone, a secondary quench is used for stage two. In this second quench, a dilute solution of urea (approximately 1%) is used to further cool and humidify the gas flow. This is an important step to assure that remaining sub-micron particulate is exposed to saturated gas, where particles can grow substantially through condensation.

Inside the scrubber vessel, several dual-orifice conditioning (DOI) trays can be installed to further condition the gas stream (stage three).

Multiple parallel MMV tubes are installed vertically on a diaphragm in the vessel (stage four). The diaphragm forces the gas flow to accelerate through the tubes. Each MMV tube includes a converging conical section (the inlet) where the gas is accelerated to throat velocity and a cylindrical throat. The diffuser outlets of the MMV tubes are aerodynamically designed to reduce the overall pressure drop by slowing down the gas and recovering the energy. In the MMV tubes, gases interact with the particulates and droplets twice (acceleration and deceleration).

Downstream of the MMV stage, ammonia acidic scrubbing takes place (stage five). The DOI tray is flooded from above with acidified water and the acid flowrate is controlled by a pH measurement. Typically, sulfuric acid or nitric acid is used to react with NH₃, forming an ammonia salt. The remaining suspended water droplets are removed from the gas stream in the mist eliminator before the gases leave the scrubber. Fresh (clean) water is continuously sprayed on the mist eliminator to catch and wash away dirty particles (stage six).

An optional wet electrostatic precipitator (WESP) can be integrated on top of the MMV scrubber to further reduce overall emissions. The basic operating principle is that particles acquire a charge. As the charged particles continue to flow through the electric field, the field forces them towards the walls of the tubes. Because the surfaces are wet, when a charged particle hits the wall, it adheres to the wet surface for washing into a collection hopper.

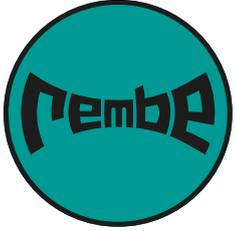
Ammonium salts reworking processes

Processing the salts generated in the ammonia removal section is often a problem. Several options have been developed to avoid having extra waste streams during the required emission control process.

In plant sites where nitric acid is not available, sulfuric acid is used as a scrubbing agent. It is widely available and contains sulfur, for which there is increasing demand as supplementary plant nutrient. The liquid urea ammonium sulfate (UAS) needs to be recycled and concentrated in order to have the salt leaving the granulation plant in a solid form after combination with melt urea. Ammonium nitrate (AN) also can be reworked by feeding it to a UAN or AN plant. Product AN preferably leaves the plant as an aqueous solution, which is then sent to a neighbouring AN/UAN plant for further processing.

In a standard configuration, the AN solution is not recycled to the urea granulation plant. For this reason, the final urea product will be pure.

A specific configuration of the industrial park can lead to a certain level of integration between the plants (Figure 6). Based on how the liquid and the gas waste streams are integrated within the plants, two different concepts are described:



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Figure 4. The Shahjalal melt and granulation plant.

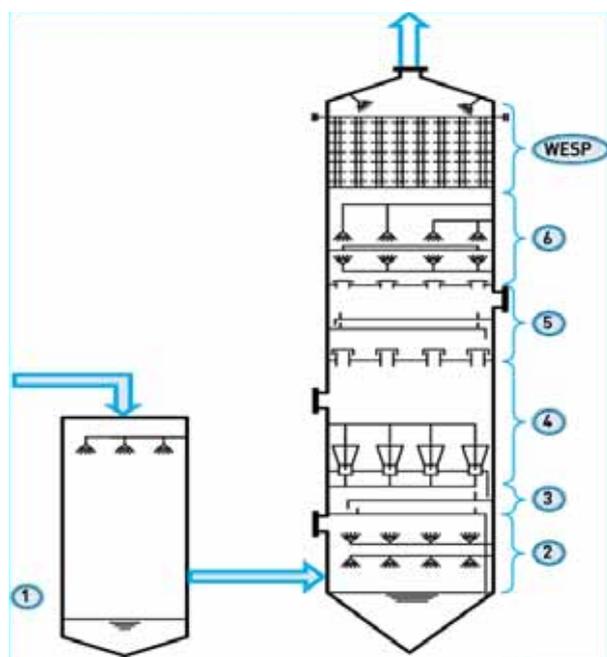


Figure 5. A sectional view of the scrubber design with the six stages indicated.

- Liquid waste stream integration.
- Liquid and gas waste streams.

In the first concept, acidic salty condensate from the off-gas treatment section of the AN/UAN plant is used in the scrubbing system as make-up water, reducing the amount of process condensate needed. Conversely, a recycle from the granulation is routed into the AN/UAN plant. In that way, no liquid waste streams are sent to battery limits.

In the second concept, a further simplification is introduced that completely eliminates the emissions coming from the AN/UAN plant. The idea is to route the ammonia off-gas that is coming from the AN/UAN synthesis section directly to the scrubbing system of the granulation plant. This solution leads to an efficient integration with a single emission point for all the plants.

Modular UAS granulation/multi-product granulation

Because of the increasing demand for granulated urea containing sulfur, Stamicarbon has developed a flexible modular process for the

production of granulated urea that contains higher ammonium sulfate concentrations.⁶ While sulfur can be made available to plants as sulfate leaching or elemental sulfur, Stamicarbon has given priority to the development of ammonium sulfate as a sulfur source instead of elemental sulfur for the following reasons:

- Ammonium sulfate is a non-flammable substance, whereas elemental sulfur is flammable substance in both solid and liquid state.
- Ammonium sulfate is non-explosive, whereas elemental sulfur dust is explosive.
- Elemental sulfur is a good electric insulator and has the potential to build up electric charges by triboelectric charging.
- Solid elemental sulfur sublimates easily and will be deposited as scale on cold surfaces, such as the inside of off-gas ducts and equipment. Ammonium sulfate does not have that property.

Elemental sulfur is practically insoluble in water, or aqueous (urea melt). To dissolve elemental sulfur in urea and keep it in solution, an additive is needed, which will contaminate the rest of the process equipment and creates foam formation in the granulator scrubber.

The properties of elemental sulfur make its application in a urea granulation plant less desirable. It is more complex, more difficult to develop and engineer, and more expensive. According to agronomic studies, bacteria needs a minimum of two years to convert elemental sulfur to ammonium sulfate, so it becomes available for take-up by plants.

In the UAS granulation process, four main modules can be distinguished (Figure 7).

Suspension module

Granulation is suitable for UAS production with an ammonium sulfate content of 0 – 50 Wt%. The additional solid ammonium sulfate is homogenised, milled (to a size of less than 1 mm), and suspended in urea melt. If the dosed ammonium sulfate concentration is above its solubility, the UAS suspension is fed to compartments one and two of the granulator. Moreover, to give them a smooth surface as an end product, a solid-free UAS melt is fed to compartment three of the granulator.

Granulation module

UAS granules are produced using the standard granulation process.

Evaporation module

The liquid effluent from the scrubber is concentrated and recycled back to the melt feed to the granulation module. The process is optimised to prevent fouling in the evaporation section.

Scrubbing module

Off-gases from the entire granulation plant are cleaned and treated as previously described. The first-built UAS plant has almost reached mechanical completion and is expected to commence in 2018. This project covers the entire scope of the UAS technology. The same modular granulation process can be used to add many other macro and/or micro-nutrients (including zinc, boron and copper) to produce urea granules.

Dry flashing

In a classical urea finishing section, the heat of crystallisation is dissipated into cooling air. The amount of air required for this cooling is vast, and cleaning is a costly and energy-intensive process since concentrations are relatively low. Dry flashing is a newly developed

process that opens up the possibility of a practically zero emission dust and ammonia finishing technology for fertilizer grade urea (Figure 8).⁷

In the urea-water phase diagram, there is a pressure and temperature region where a urea solution will spontaneously split into two phases: solid urea and water vapour. This can be performed in a pressure-controlled flash vessel.

The urea produced in this process step is in the form of a fine powder (average particle sizes are a magnitude of 100 – 150 µm), which needs to be further treated into the final shape required. Condensate and overhead vapours from the process system can be treated in the existing melt plant and will not lead to any extra ammonia or urea losses.

In this process, urea solution is concentrated to the desired extent in a pressure and temperature controlled evaporator. The urea solution from the evaporation step is introduced into the flash vessel, where the pressure is kept at a sufficiently low sub-atmospheric pressure by means of a dedicated vacuum system. The solid urea that is formed is extracted from the flash vessel through a vacuum lock and is transported for further processing.

A contact condenser is introduced to deal with urea particles that are entrained in the water vapour leaving the flash vessel overhead. The overhead vapours from both vacuum systems (evaporator and dry flash vessel) are combined and processed in the atmospheric absorber of the urea melt plant.

A further advantage of this process concept is that the produced urea contains significantly less biuret than conventionally granulated urea. Because the urea melt solution does not have to be concentrated to the same extent as a conventional granulation, the final product contains low biuret. This makes the process a perfect tool for producing diesel engine fluid (DEF) or cattle feed. The solid urea product can be transported to the DEF reseller or distribution centre without a huge amount of water. In the DEF distribution centre, the urea is dissolved at the concentration needed.

The ultimate goal of this dry flashing technology is to produce fertilizer grade urea. The produced urea should be of a comparable quality to the currently available granulated product. Stamicarbon expects that such a new urea finishing process for industrial scale fertilizer application will be launched to the market at the end of 2018.

Conclusion

Finishing technologies have been used in urea production for a long time. Changing markets and business situations, as well as increasingly strict environmental requirements and legislation, have all been drivers for Stamicarbon to innovate its finishing processes. **WF**

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Figure 6. Available plants for ammonium nitrate reworking.

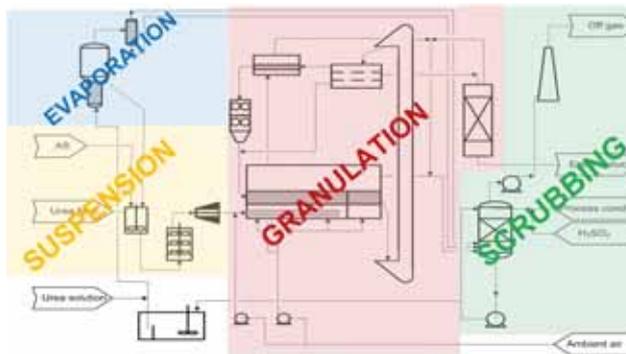


Figure 7. A modular analysis of the Stamicarbon UAS fluid bed granulation plant.



Figure 8. Solid urea from a dry flash.

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THE BIGGER PICTURE

Gelmer Bouwman, Kreber, the Netherlands, considers how efficient prilling towers can help to cope with the growing global demand for fertilizers.

The global population is growing, which means that there is a concurrent increasing demand for food. There is already difficulty in keeping food production up with current demand, yet fertilizers can enhance the growth of food and, therefore, aid in meeting global food demand. Fertilizers such as urea and ammonium nitrate are produced at large capacity to reduce their market price. Expanding the production of these fertilizers comes with the cost of increased emissions. The recently signed Paris climate agreement demonstrates a growing concern over global warming, especially the effect of emissions. This will result in increased efforts and demands to significantly reduce emissions. It is expected that in the near future only well-integrated equipment will be employed.

In recent years, Kreber has changed its focus from merely producing equipment, towards developing knowledge. The company's R&D department collaborates with Delft University of Technology to work on the development of fundamental knowledge with respect to topics that include, but are not limited to, vibrating buckets, stationary prillers, bulk solids handling equipment, and emission reduction solutions; all within the scope of prilling towers.

Why prilling towers?

A prilling tower is a finishing section in the fertilizer production process. Even with considerable efforts at developing granulation technology over the past decades, prilling is still the most economically viable finishing technology. A natural draft prilling tower has low operational expenses, as well as limited capital expenses. This is the reason why most new towers that are built in China, India, and Russia are designed with

natural convection. However, there are several limitations and problems observed with prilling towers, such as the maximum achievable prill size and the control of emissions.

Prill size

The size and size distribution of prills is a topic of much debate. For the bulk mixing of fertilizers, the size should be similar to that of the other fertilizers, which is uniform in most cases. A uniform distribution makes the operation of finishing processes easier as there will be less caking and more predictable cooling properties. On the other hand, most farmers prefer a wide range of prill sizes since this enables the efficient spreading of the fertilizer. Also, varied particle sizes result in a bulk density increase, which is favoured for shipping. The final choice is dependent on the required prill properties. Notwithstanding, dust formation and its emission is undesired.

To reduce the amount of dust in the final product and limit the formation of satellites (droplets that are typically 100 μm in diameter), Kreber is exploring possibilities to eliminate the scraper at the bottom of the tower and develop vibro technology for prilling buckets. The scraper, which collects the prills, however gently, causes collisions and abrasion between the prills, which results in dust formation. Hence, the need for an innovative prill collection system. While applying vibration to a prilling bucket is known to enhance the uniformity of prills, in a rotating high capacity bucket there are several obstacles that need to be overcome. Currently, a prototype bucket has been designed and will be tested in an industrial environment in collaboration with a launching customer.



Figure 1. An overview of global urea finishing technologies. At least 68% of urea production facilities make use of a prilling tower, which illustrates the importance of emission reduction.¹

Emissions

The emissions from free convection prilling towers are excessive and are difficult to control. The amount of emissions is at least an order of magnitude larger than the emissions in prior process steps in fertilizer production. The emissions considered here are dust and ammonia. For many processes, filters and scrubbers are used to treat the off-gas. However, with free convection, it is difficult to install filters since the driving force (density difference) is not large enough to overcome the relatively large pressure drop over a filter.

Emissions regulation in Asia are less strict than those in the US and Europe. It is, therefore, possible to construct low-cost prilling towers in less regulated countries. However, with recent developments on climate change, regulations worldwide are expected to become tighter. In order to limit emissions and reduce impacts on air quality, it is important to develop a solution that is compatible with free convection prilling towers. Kreber has conducted an intensive study with Delft University of Technology to explore innovative possibilities.

The goal of the study was to develop a system that reduced emissions from a free convection prilling tower, while meeting the following design objectives:

- Low operational expenses.
- Low capital expenses.
- Low complexity.
- Good prill quality.
- Long lifetime.

There are several difficulties faced when treating emissions. First, the dust and ammonia concentration in the air stream is low. Next, the amount of air flowing through the prilling tower is large. Furthermore, introducing a treatment section results in a pressure drop, which needs to be overcome. All three aspects encumber the design of a feasible system. In order to design an innovative system, companies must have a proper understanding of the problem, which includes knowledge on the source and composition of the emissions.

Ammonia emission

The ammonia (NH_3) that is present in the off-gas from prilling towers has multiple sources. Ammonia can be found in the feed towards the prilling bucket since it is a

reactant of the production process. The main fraction of unreacted ammonia is recovered by evaporation. Melt is pumped from the evaporator to the prilling bucket. Here, the ammonia concentration is low and the temperature is typically just above the melting point of urea, approximately 135°C. These conditions favour the hydrolysis of urea (reverse formation of urea) and the formation of biuret. Ammonia is a product of both reactions. Other reactions, such as the formation of triuret, are also present, albeit very limited due to the process conditions and the low residence time.²

The ammonia can enter the gas phase when urea is in the liquid phase. It enters the prilling bucket and in the first part of cooling, i.e. when a droplet of urea melt is formed. When the droplet reaches the melting point, a thin shell of the prill is solidified. At this point, free ammonia is captured within the prill. The concentration of ammonia is reduced when it reacts with isocyanic acid (HNCO) to form dust, as described below.

Free ammonia that is present in the molten urea is either not recovered by evaporation or formed due to biuret formation. It enters the gas phase from urea in the liquid phase and is removed from the gas phase when it reacts with HNCO to form dust. The concentration of ammonia in the off-gas from prilling towers is typically low, in the order of 100 ppm. However, due to the large volume of air moving through prilling towers, ammonia emission can exceed 2 tpd.³

Dust emission

Dust particles present in the off-gas from prilling towers have different sizes. The difference in size is due to different mechanisms by which the dust particles are created. Particles in the order of magnitude of 1 µm, can be formed by either deposition of HNCO and ammonia or deposition of urea vapour. The vapour pressure of liquid urea at 135°C is noticeable, i.e. urea vaporises under these conditions. Larger dust particles can be formed by either solidification of satellite droplets at the top of the prilling tower or due to prill degradation at the bottom of the prilling tower, as a result of collisions and abrasion of prills.⁴

Table 1 presents a summary of dust formation in a prilling tower. The values are rough estimates and give an indication of different types of dust that are formed. The size resembles the order of magnitude of the dust particles. The fraction of dust will differ for each tower design. For example, the fraction of satellites is dependent on the type of bucket.

Separation of flows

The majority of dust emissions are formed in the top section of the prilling tower, particularly near the prilling bucket. The polluted stream leaving the tower is very dilute due to vast amounts of air passing through the prilling tower. This dilution makes it difficult to design a feasible system to remove pollution from the stream. The proposed concept has two streams leaving the prilling tower; one dilute stream and one stream with a high dust load and high ammonia concentration.³ The concentrated stream funnels the air from around the bucket to a

treatment section, while the other stream conducts the main portion air, which contains limited amounts of pollution.

Separating the flow enables a feasible design of a treatment section for the concentrated emissions stream. The separation of flows is realised by placing an exhaust in both the centre and outer circumference of the prilling tower. This design should allow concentrated air to exit through the centre exhausts. The concentrated stream can be treated by a cleaning section after which it is vented into the atmosphere. The diluted air leaves through the outer exhausts of the tower and is directly vented.

Treatment section

Removing the ammonia from the gas stream can be accomplished with an adsorption column and an option for the adsorbent is biochar. When the adsorption capacity of the biochar is reached, it can be sold as a fertilizer to the local market. Other adsorbents, such as activated carbon, can also be used for the adsorption column, meaning that the activity of the adsorbent can be enhanced by activating the adsorbent with phosphoric acid.⁵

Dust removal from prilling towers is a complex task due to the range of the particle sizes found within. The majority of the dust particles can be measured in microns. Plenty of systems are available to remove dust particles from the dust laden air streams. Both the ammonia and dust treatment in off-gases will result in a pressure drop that has to be overcome by either a compressor or fan. An innovative idea is to make use of bladeless fans on the much smaller separated and concentrated air streams. This type of fan will require lower maintenance since it is not possible for dust to accumulate on the blades. However, because it is a novel technology, industrial sized applications are not yet available, which it may initially result in higher capital expenditures.

Prevention instead of treatment

A different concept to reduce emissions from a prilling tower is to inject an additive into the urea feed stream. The central idea of this concept is to bind free ammonia at the source of origin, something that can be done with an acid to yield a salt. Challenges with this design are: choosing which acid to inject; choosing where to inject the acid; and knowing what the effect of acid injection will be on both emissions reduction and prill quality. The technology of injecting acids looks promising when addressing the design objectives presented above, and on the results from the literature study performed.⁵

Conclusion

With the desired long-term increase in production capacity of fertilizer, reflecting on reducing emissions is unavoidable. Three options for emission reduction have been presented in this article:

- Elimination of dust at the source to prevent satellite droplet formation in molten stage and omitting the use of a bottom scraper.
- Treatment of concentrated emissions with a filter and an adsorption column.

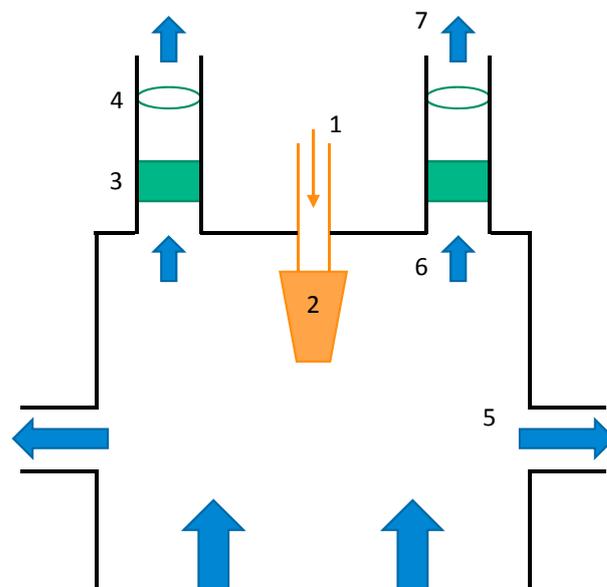


Figure 2. A schematic illustration of the topside of a prilling tower. Melt (1) entering the prilling bucket (2) is distributed in the top section of the tower. On top of the tower, a treatment section is placed consisting of a filter (3) and a bladeless fan (4). The main fraction of cooling air leaves the tower untreated through an exhaust (5). The polluted air (6) is sucked through the filter and vented to the atmosphere (7).⁵

Table 1. Summary of dust formation in a prilling tower⁴

Dust type	Size (µm)	Fraction (%)
Urea vapour deposition	1	50
HNCO and NH ₃ deposition	1	20
Satellite droplets	100	5
Prill degradation	100	25

- Prevention of emissions using the injection of an acid in the melt before prilling.

The separation of flows and introduction of relatively small bladeless fans are innovative solutions that may be an alternative to the application of conventional scrubbers and will be subject to further research.

Global needs and requirements, as well as market demands, give insight into which knowledge adds most value. Kreber has a strong desire to continue developing and increasing its knowledge base. The company is keen to collaborate as working towards a better future should be a collaborative effort. **WF**

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GRANULATION: AN EVOLVING INDUSTRY

NICK RECKINGER, FEECO INTERNATIONAL, INC., USA,
COMPARES TRADITIONAL AND NOVEL APPROACHES
TO GRANULAR FERTILIZER PRODUCTION.



Since the advent of the Tennessee Valley Authority's (TVA) approach to granulation in the 1950s, widespread, commercial-scale use of the process has become the backbone of the fertilizer industry. And while many of the principles around granulation have remained the same, some variations and novel approaches to the process have been developed. As companies look for increasingly efficient ways to produce even better products, R&D efforts are ongoing.

Benefits of granulation

The use of a granular product over a material in 'raw' form offers a number of benefits that have allowed granulation to become a staple in agriculture. Among these benefits:

- Granulation mitigates many of the issues associated with dust, ultimately improving handling, transportation and application. Product loss via dust is also reduced, improving the bottom line and providing more predictable results.
- Product appearance and characteristics are improved, creating a marketable premium product.
- Caking, lump formation and segregation are reduced with a granular product.
- Granulation can be used to create an all-in-one granular product, where each individual granule contains the desired nutrient formulation.
- Granulation can be a highly flexible process, allowing various additives (such as micronutrients

or organic matter) to be incorporated into the product.

Factors pushing growth in granulation

While the benefits of granulation have facilitated its widespread use, a number of factors have spurred a renewed interest in the process in recent years.

Use in precision agriculture

Precision agriculture is an up and coming technology whereby fields are mapped out using geolocation technology to pinpoint precise nutrient requirements within a given area to address in-field variability. Variable rate technology (VRT) is used for precise application of the nutrients to meet the fluctuating needs throughout the field. A granular product is a key



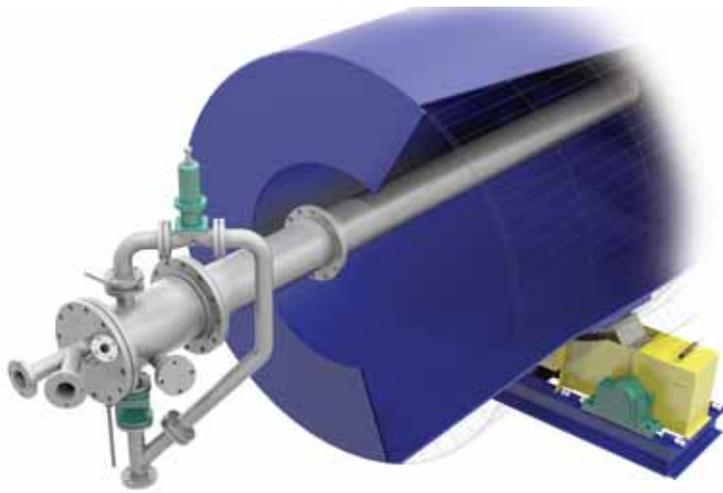


Figure 1. A 3D model of a FEECO pipe reactor that is integrated into a granulation drum.



Figure 2. A FEECO rotary dryer is prepared for shipment.

component in the success of this practice as it allows for measured and accurate dispensing of the nutrients.

Customisable product formulations

As more is understood about creating a sustainable relationship between crop production and nutrient delivery, a one-size-fits-all approach is no longer acceptable, causing demand for products that are tailored to a specific application, crop or region to increase. This has fertilizer producers creating customised products through granulation to meet their specific needs.

A need for sustainable solutions in manure management

Large scale farming operations and centralised animal feeding operations have become the agricultural norm in recent years. This has farms producing more manure than they can economically and sustainably manage. Paired with increasing attention around nutrient runoff, farmers are in need of a better approach to manure management. This has many looking at granulation as a possible solution; manure can be granulated into a dry product, reducing transportation costs, aiding in nutrient management, as well as improving on-farm logistics and economics.

Approaches to granulation

There are many approaches to producing a granular fertilizer, with the most common of them outlined below. Fertilizer granulation can be divided into two major categories: those that utilise binder and movement to form granules (non-pressure), and those that utilise pressure to form granules (pressure agglomeration).

Non-pressure granulation techniques

Non-pressure granulation, also referred to as tumble growth or agitation agglomeration, utilises a binder combined with a tumbling action to promote granule formation. Non-pressure approaches typically yield a product in the form of a rounded granule.

Rotary drum granulation

The major equipment utilised in this approach includes a granulation drum (granulator), rotary dryer, rotary cooler, coating drum, and screens. The process is typically used for inorganic fertilizer products, such as nitrogen, phosphorus and potassium (NPK), MAP, DAP, AS, TSP, SSP, sulfur enhanced products, etc.

The rotary drum is the basis of this fertilizer granulation process. The approach may include a chemical reaction or no reaction, and along with a tumbling action, the granulator can create various grades of fertilizer.

Feedstock components for this process can be powdered raw materials, ammonia, acids or pre-reacted materials from a pre-neutraliser. The materials, along with recycle material, are fed into the granulation drum where the rotation of the drum imparts a tumbling action on the bed of material to form granules. The 'green' (wet) pellets are then dried in a rotary dryer to remove moisture and polish the granules before being transferred to a rotary cooler to stop any ongoing reaction and to cool the product for subsequent handling, storage or bagging. The material is screened prior to product loadout where the undersize and crushed oversize materials are sent back to the granulator. Often, a coating drum is added before product storage to minimise dust and caking for shipment.

Key advantages of the rotary drum granulation method include it being a time-tested, simple approach that is flexible. Moreover, the product has significantly reduced dust compared to those produced via pressure techniques.

Pipe reactor granulation

The major equipment utilised in this approach includes a granulation drum, pipe reactor (or cross-pipe reactor), rotary dryer, rotary cooler, coating drum, and screens. Typical applications of the process include: ammonium sulfate, high reaction NPK grades, and some novel organic-based fertilizers.

Pipe reactor granulation is similar to the drum granulation approach. However, it employs a cross-pipe

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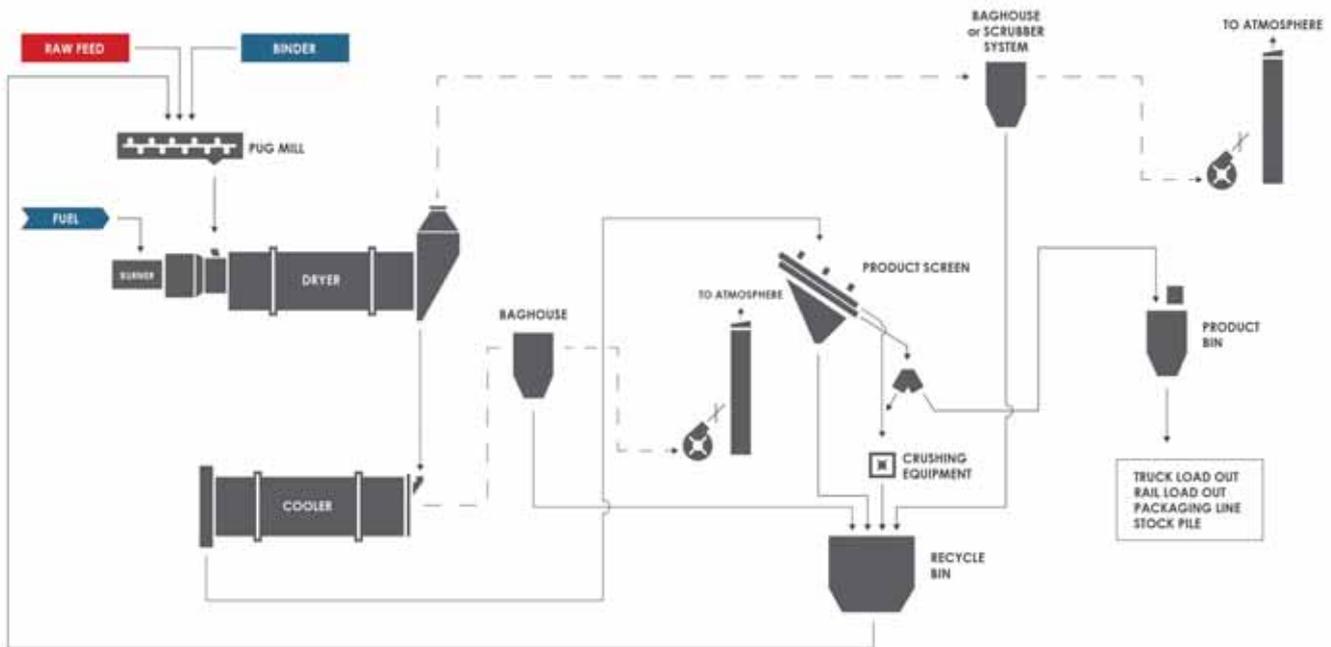


Figure 3. A simplified mixer-dryer granulation system for manure.



Figure 4. Manure granules created via granulation in the FEECO Innovation Center.



Figure 5. Ammonium sulfate granules created in the FEECO Innovation Center.

reactor to facilitate the reaction of the acid(s) and ammonia. The pipe reactor is integrated into the granulation drum, as shown in Figure 1. For retrofits, this setup can replace the pre-neutralising step.

While a pipe reactor is not required in any system, in the right setting, it can offer significant energy savings because the captured heat of reaction helps to dry the material, in turn reducing the energy required for the dryer.

The method works by feeding phosphoric or sulfuric acid into one side of the pipe while gaseous or liquid ammonia is fed into the reaction chamber. This results in a hot 'melt' of either ammoniated phosphate or ammonium sulfate. Scrubber water is also added to control the reaction. The melt is sprayed onto the bed of material in the granulator to form the granules and much of the moisture is flashed off. The wet pellets are then dried in a rotary dryer to remove moisture and polish the granules. As in the previously described granulation drum approach, cooling, screening and coating typically follow the dryer.

A key advantage of this method is that it has potential for significant energy savings. Moreover, the process can be retrofitted into existing systems easily, it only uses liquids and gases for feed materials, and, again, the product has significantly reduced dust compared to those produced via pressure techniques.

Mixer-dryer granulation

The mixer-dryer approach is similar to the drum granulation method, but instead of a drum, this process relies on a mixer to granulate the material. This granulation method requires a pug mill (paddle mixer), rotary dryer, rotary cooler, and screens. It is typically used for organic-based fertilizers.

Feedstock, binder and dried recycle are fed into the pug mill at a continuous, specified rate. Here, granules are formed as they move down the length of the mixer. After the necessary retention time, the 'green' pellets are carried via conveyor to a rotary dryer, where the moisture content is reduced. Again, the tumbling action



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HTHA AUBT and High Frequency Phased Array Inspection

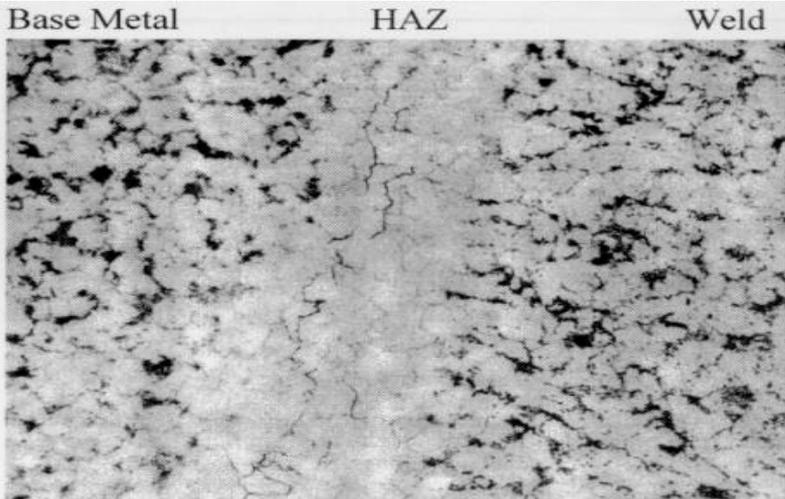
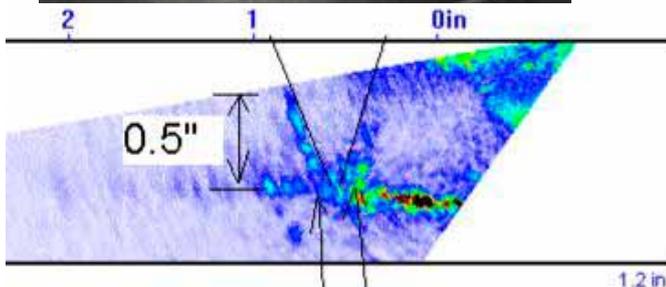
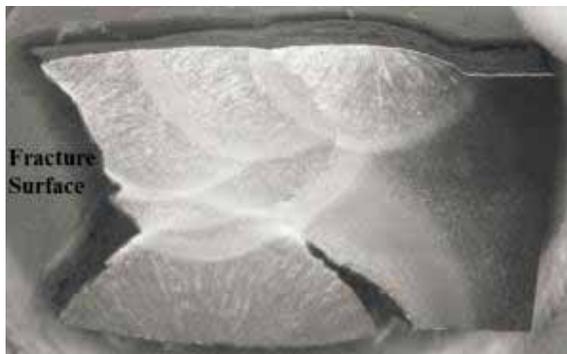
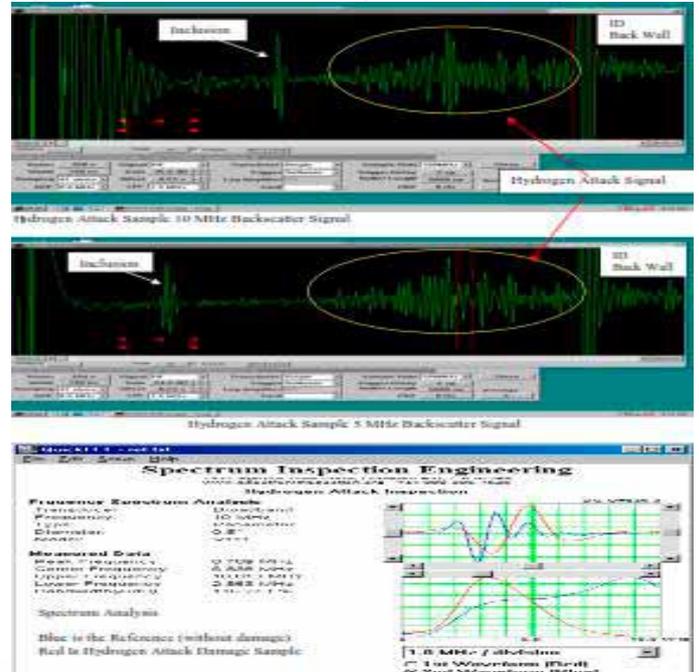
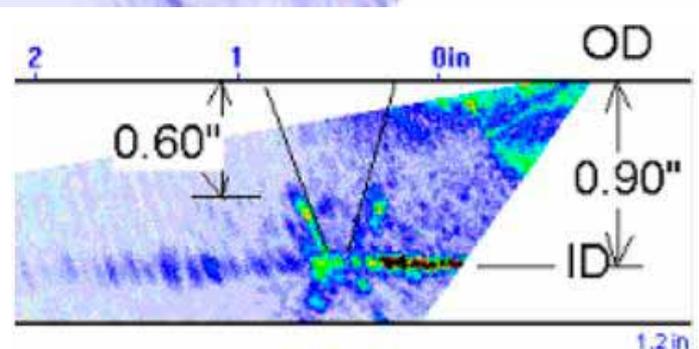
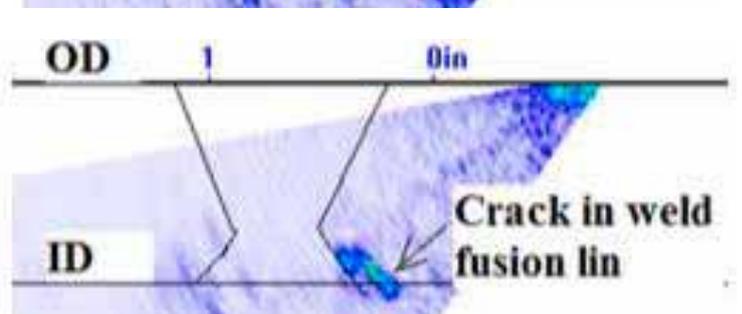
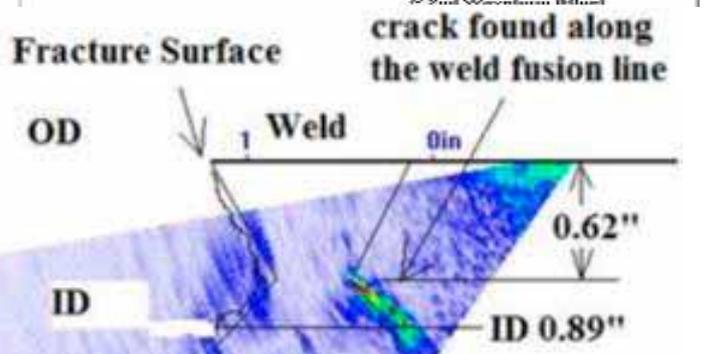


Figure 1 Localized HTHA in a carbon steel weld HAZ. The light area is the HAZ, decarburized due to HTHA. The dark, curvy lines in the HAZ are fissures caused by HTHA.



Suspected Indications follow the weld fusion line



1.2 in

of the rotating drum further rounds and polishes the pellets, and cooling and screening typically follow the dryer.

This process allows for a highly customisable product that has significantly reduced dust compared to those produced via pressure techniques.

Disc pelletiser granulation

The disc pelletiser method of granulation employs a disc pelletiser, pin mixer or pug mill (recommended), and a rotary dryer. It is typically used for ammonium sulfate and soil amendments as it is better suited to more finely divided solids (such as chicken litter) or when a refined product is of utmost importance.

Feed materials and binder are fed simultaneously at a continuous rate onto the disc pelletiser. As the disc rotates, the tumbling action, in combination with the binder, causes the fines to become tacky and pick up more fines as they roll along the disc and against each other, forming pellets.

Alternatively, a pin mixer or pug mill preceding the disc pelletiser can be used to thoroughly mix feedstock ingredients with the binder to create a homogeneous mixture and begin forming seed pellets. This recommended step creates a more refined pellet product, allows for improved size control, increases production and reduces binder use, as densification is a result of the action imparted by the mixer instead of additional binder.

Using this method produces a premium product with more on-size pellets. The process allows for increased throughput compared to mixer-dryer granulation and allows for a highly customisable product.

Pressure agglomeration techniques

Compaction granulation

The main equipment used in this process includes a roll compactor, a flake breaker, a polishing drum (where applicable) and rotary dryer (where applicable). Typical applications include the production of ammonium sulfate and potash.

Compaction granulation is a widely-used pressure granulation technique throughout the fertilizer industry. Unlike the non-pressure methods that create a rounded pellet product, compaction granulation creates granules with jagged edges.

Raw feed material is fed into the roll compactor. The material is fed between two counter rotating rolls, which come together to press the material into a compacted sheet. As the sheet of material moves through the rolls, it passes through a flake breaker and granulator, which breaks the sheet up into the desired size particles.

Because pressure is used to form the granules in this process, a binder is typically not needed. Though, in some cases, the addition of a small amount of binder can be helpful. As such, this is typically a dry process, meaning that a drying step is not required. However, when a binder is employed, a drying step is necessary after granulation to remove the added moisture.

In some cases, a polishing drum may be utilised to knock off any loose granule edges and round the hard edges of the material to reduce the occurrence of attrition. A glazing step may also be employed.

Key advantages of this method include lower operating costs compared to non-pressure methods and, typically, no drying is needed.

Choosing the best approach

Choosing the most effective approach for a specific material or process setting requires careful consideration of a number of factors. Some of the most important considerations are reviewed here.

Product requirements

The product requirements will often help in choosing the best suited granulation method for a given scenario. What form is the feedstock material in? Is the material capable of being processed via pressure techniques (not all materials will adhere to themselves under pressure)? Is a customised product desired? Is dust and product loss a major concern? Will the product require fast breakdown upon application? These are all things that need to be considered when choosing a granulation approach for a product.

Cost

Cost is a major factor in any decision, and with so many options, it is no less important when it comes to choosing the best granulation method for a given project. While compaction granulation requires a higher initial capital investment, it offers lower operating costs compared to non-pressure methods. Conversely, the non-pressure agglomeration methods require higher operating costs, but produce a premium product that is highly marketable.

Batch and pilot testing

One way of taking some of the guesswork out of choosing the best approach to granulation is through testing.

Having a material run through a batch and/or pilot testing facility, such as the FEECO Innovation Center, can help to define process variables, determine which method of granulation will produce the desired results, and produce a recipe for process scale-up.

Conclusion

While fertilizer granulation itself has not changed much since TVA implemented its approach, improvements and variations of the process have developed to meet more specific industry needs. Various approaches to granulation are now available, with most processes offering a high degree of customisation.

Choosing the most effective approach is a balance of finding a method that suits the product requirements and material characteristics that can be commercialised on an economic scale. For more novel products and systems, this is often determined through testing at both batch and pilot scale. **WF**



RISKY

CHRIS JONES, AUSTRALIA, AND CHARLES THOMAS, NEW ZEALAND, QUEST INTEGRITY, AND DAVID KEEN, INCITEC PIVOT LTD, AUSTRALIA, LOOK AT INSPECTION OPTIONS FOR MITIGATING HIGH TEMPERATURE HYDROGEN ATTACK.

BUSINESS

Managing the risk of damage due to high temperature hydrogen attack (HTHA) has been a major problem for the synthesis gas industries. The reasons for this are largely twofold: firstly, the likelihood of HTHA occurring has proven difficult to predict and, secondly, the detection of HTHA by inspection is problematic. Given the potential for extreme consequences in the event of a catastrophic release of hydrogen (as seen in the Tesoro Anacortes incident in 2010),¹ the need for a multifaceted approach based upon an integrated risk management plan is imperative. The risk is illustrated in Figure 1, which shows the hydrotreater exchanger that failed at Tesoro and resulted in seven fatalities. This photograph is much reproduced but remains an important reminder of the potential for catastrophe caused by unchecked HTHA damage.

This article describes recent experiences relating to damage found in equipment that may have been considered safe using traditional approaches to managing risk, as well as the inspection techniques required to detect that damage.

HTHA

The process of HTHA requires the dissolution of atomic hydrogen. This is normal as all ferritic steels operating in hydrogen at an elevated temperature and pressure will dissolve hydrogen. The higher the temperature and pressure, the greater the amount of dissolved hydrogen. Another manifestation of this phenomenon is the frequent need to 'bake-out' or heat treat steels in order to remove the dissolved hydrogen before attempting the welding of steels. The dissolved hydrogen is reactive; it will react with any free



Figure 1. Damaged heat exchanger at Tesoro.¹

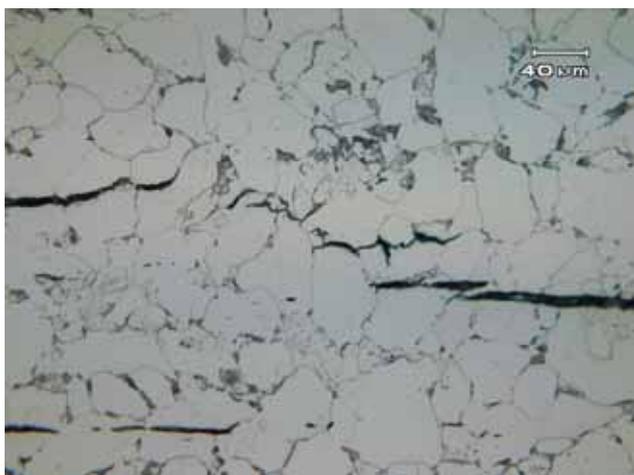


Figure 2. Example of internal fissuring caused by HTHA.

carbon or internal carbides, both of which have significant influence on material strength.

This carbon and/or carbide reaction generates methane gas. The more stable the carbides in the steel, the less likely the reaction is to occur. Consequently, more alloyed grades are more resistant to HTHA. Methane is not soluble, and forms in small pockets around internal inclusions. The localised pressures here can be extremely high, and localised high stress levels are generated leading to the formation of internal fissures and micro-cracks. Near-surface pockets of methane can lead to blistering. The formation of micro-fissures and the loss of carbon results in serious reductions in strength, ductility and toughness. The reduction of these material properties will ultimately result in the component being unable to withstand the normal service loads, and catastrophic failure will occur (Figure 1). Figure 2 shows an example of the internal micro-fissuring caused by HTHA, which must be either prevented or detected by advanced inspection.

The Nelson Curves

For many years, significant confidence has been placed in the Nelson Curves, as described in API 941, to predict the likelihood of HTHA.² These curves are based on reported performance and have been subject to a number of significant changes as new experiences come to light. Figure 3 shows the Nelson Curve from API 941. This set of curves represents the core information in the API standard. In the 1990s, there was a

growing number of cases of HTHA damage occurring in carbon-½molybdenum steels. As a result, the carbon-½molybdenum curve was removed from the API 941 guideline, thereby placing a large volume of carbon-½molybdenum equipment in limbo alongside the recommendation that the material should be treated as carbon steel as far as its resistance to HTHA was concerned. Due to this API 941 addendum, a large number of affected equipment already in-service and designed to the original carbon-½molybdenum curve was operating above the carbon steel curve.

An attempt was made to rationalise the experience with carbon-½molybdenum steel by providing different risk factors for annealed and normalised material.³ The implication is that the metallurgical condition of the material, in this case the form and stability of the carbides, was significant. There is, however, no reason to believe that such an effect is unique to carbon-½molybdenum steel or indeed parent metal as opposed to weld and HAZ material. An attempt to quantify risk factors using a parameter P_v dependent on pressure, temperature and operating pressure was included in the 2008 version of API 581,³ but this was removed from the 2016 version.⁴

The second major change occurred in the wake of the Tesoro incident and the subsequent Chemical Safety Board Report.¹ The investigation concluded that failure of the exchanger occurred due to HTHA in a carbon steel pressure shell that was operating below the Nelson Curve. Significantly, the exchanger had not been post weld heat treated, and all the observable damage was present in the heat affected zone. API 941 contains comprehensive information concerning the influence of stress on the risk of HTHA. The 2016 edition contains the first attempt to quantify this by introducing a new curve for carbon steel that has not been post weld heat treated.

As in the case of metallurgical condition, there is no reason to believe that the effect of stress is unique to one particular grade of steel. However, it is very likely that more highly alloyed grades will be post weld heat treated. As API 941 details, the risk of HTHA in steels that operate within the ASME allowable stress limits, and below the Nelson Curves, is negligible. The influence of stress does need to be taken into account when severe stresses (such as weld residual stress, thermal stress or high piping loads) exist. Being aware of the existence of such stresses is an essential part of any risk management plan.

Despite these limitations, API 941 and the Nelson Curves remains the principal guide to managing the risk of HTHA. It is an experience-based document and continuous modification in the light of new information is to be expected. By themselves, the Nelson Curves do not guarantee freedom from HTHA.

Risk assessment

As noted above, the API 581 approach to establishing risk levels using a single quantifiable parameter P_v has been removed from the recommended practice.³ The 2016 version has adopted a very conservative approach, particularly for carbon and carbon-½molybdenum steels. For these steels, the current version API 581 assigns high susceptibility for any component operating above 177°C (350°F) with a hydrogen partial pressure exceeding 0.345 MPa (50 psia). This is very conservative relative

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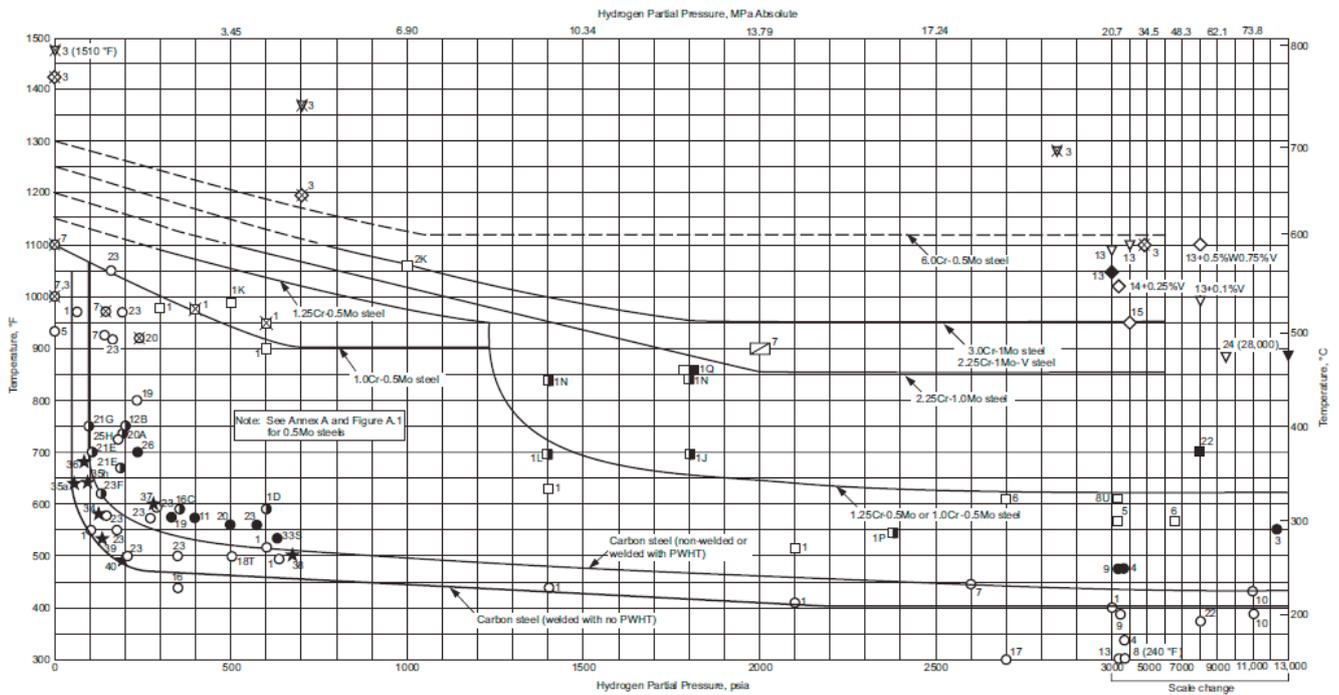


Figure 3. Operating limits for steels in hydrogen service to avoid HTHA.²

to the API 941 Nelson Curves for both post weld heat treated and non-post weld heat treated carbon steels. It is acknowledged, however, that this is an interim measure while a more quantitative method is under development.

For low alloy steels, the calculation of risk factor has also been simplified through the acknowledgement that the Nelson Curves do not represent an absolute measure of risk, but that assigning a risk level based upon the margin by which any component operates below the Nelson Curve is advisable (Figure 4).

This high level of conservatism on the one hand, and scepticism around the reliability of the Nelson Curves on the other, has led some owners to develop their own methodologies. An example approach is summarised below.

Step 1: identifying susceptible equipment

All equipment in hydrogen service is identified and categorised as:

- High susceptibility: operating on or above the Nelson Curve.
- Low susceptibility: operating below but within a 50°F and 50 psi below the curve.
- No susceptibility: operating more than 50°F and 50 psi below the curve.

Step 2: undertake risk assessment

For all equipment that is identified as being susceptible to HTHA, a risk assessment is performed. Using standard risk methodologies, a risk matrix can be generated. The factors to be taken into account for HTHA include:

- Temperature.
- Hydrogen partial pressure.
- Material of construction.
- Exposure time.
- Stress.
- Cladding.
- Previous failures.

- Inspection history.
- Heat treatment.

Step 3: develop a risk management plan

A primary objective would be to reduce the risk for those assets in which the risk is assessed to lie within the 'high risk' (red) area of a risk assessment matrix. This may be achieved in the short-term to allow operation to the next scheduled outage by reducing operating conditions, usually by adjusting temperature. Long-term solutions involve:

- Developing robust inspection test plans (ITP). This may include destructive testing.
- Enact inspection to be conducted at scheduled outages.
- If HTHA is found, the consideration of equipment replacement with upgraded metallurgy is advisable.

Step 4: integrate with asset integrity management plan

It is critical for ongoing risk management that knowledge and operational limits be understood and acted upon within the wider organisation. Key issues that must be addressed include:

- Knowledge, training and knowledge management: it is essential that the significance and risk of HTHA (and indeed all potential damage mechanisms) are understood throughout the organisation.
- Establishment of integrity operating windows (IOWs): the risk of HTHA is strongly influenced by operating conditions. Establishing and implementing IOWs is required to ensure continuing operation. This should include reporting protocols so that the impact of any excursion can be assessed.
- Plant standard operating procedures (SOPs): the impact of HTHA should acknowledge the significance and risk of HTHA and the control measures that are in place to manage that risk.

- Management of change (MOC): robust MOC procedures are required to ensure that any changes to operating conditions do not impact negatively on the reliability of the plant.

Inspection

The risk management plan for each piece of equipment that is deemed susceptible to HTHA inevitably calls on inspection to determine the presence and extent of HTHA. The ultimate control of HTHA is to replace at-risk equipment. This is usually a costly solution and justification would normally require evidence that HTHA damage had indeed occurred. API 941 contains commentary on the various candidate inspection techniques and concludes that no single inspection technique is ideally suited to detection of HTHA damage, particularly in its embryonic stage when micro-fissuring is just beginning to form.²

Ultrasonic inspection (UT) has been found to have the best chance of detecting HTHA. Four generic types have been used, as listed below:

- Backscatter techniques.
- Velocity methods.
- Attenuation.
- Spectral analysis.

These are illustrated schematically in Figure 5. Backscatter UT involves irradiating the test material with moderately high frequency ultrasound and measuring the backscatter noise that is reflected from HTHA. This can be used to detect HTHA and also to provide an estimate of the concentration and through thickness extent of damage.

Velocity methods make use of the fact that the altered microstructure from HTHA produces material with different shear wave ultrasound velocity than the unaffected material, whereas compression wave ultrasound velocity is relatively unaffected.

The backwall reflection is essentially a wall thickness test: the extent to which the signal is attenuated by the presence of HTHA can be used to characterise the extent of HTHA and differentiate HTHA from other forms of damage.

An extension of this technique is spectral analysis in which the dependence of attenuation on the frequency of the ultrasonic signal is analysed. The higher frequency spectrum from the reflected backwall of a broad band ultrasonic signal is more extensively attenuated. Such analysis can help differentiate between HTHA and other apparent damage, such as embedded defects. The success of this approach is dependent on the sampling technique, which can be impaired by the following factors:

- Non-parallel material geometry.
- Thin wall tube.
- Austenitic clad material.
- Localised damage at weldments.
- Inherently attenuative material.

None of these techniques are ideal, and an inspection methodology has been developed that uses all four. This is the advanced ultrasonic backscatter technique (AUBT). In this methodology, backscatter methods are used as screening tool to locate damaged areas, and based upon the backscatter pattern observed, one or more of the other UT techniques are



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Table 1. Results of the inspection programme	
Item	Result
Equipment items assessed susceptible to HTHA	75
Equipment items inspected for HTHA	36
Equipment items confirmed to have HTHA	22 (61% of inspected items)
HTHA identified by AUBT	1
HTHA identified by AUBT with qualified operator	18
HTHA confirmed by metallurgical sample	2

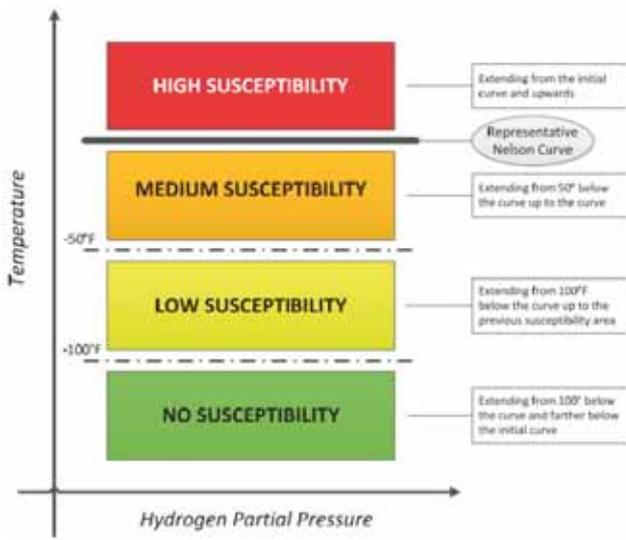


Figure 4. An example risk assessment for low alloy steels in an HTHA environment (API 581).⁴

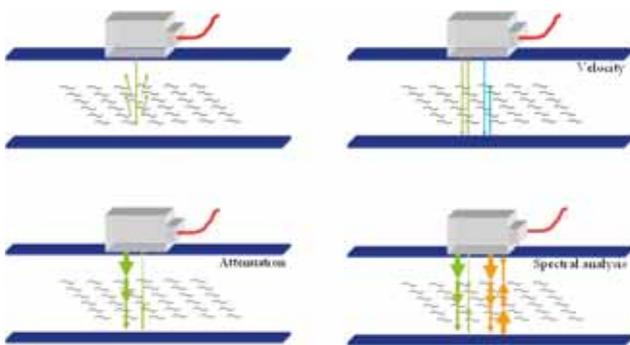


Figure 5. Schematic illustrations of UT inspection techniques for HTHA.

employed to qualify the presence of HTHA damage. A rigid approach to the characterisation of the damage from the ultrasonic methods, in all situations, will not provide a successful result. Success depends on the operator's ability to assess, select appropriate methodologies and analyse the data.

The AUBT technique and its variants have been used extensively in the integrity management and inspection programmes at Incitec Pivot Plants (IPL). The results are summarised in Table 1.

Historical analysis of HTHA inspection at IPL has indicated a strong dependence on the knowledge and experience of the operator performing the inspection. AUBT inspections that have been contracted without first qualifying the inspection

personnel have been largely unsuccessful. However, the inspections performed by inspectors in which the owner had confidence have proven very successful. In this context, a 'qualified' inspector is one that has verified that the methods, and procedures employed during the inspection have a very high chance of detecting HTHA, if present. It does not mean that the inspector has a certificate confirming attendance to an HTHA training course.

Given the absence of any single or reliable UT technique to detect HTHA, the findings at IPL have been that the experience and the knowledge of the inspector is paramount to the success of the inspection programme. The importance of a robust asset integrity management programme was noted previously, and a number of the activities that go to make such a programme were highlighted. The results of the HTHA inspection programme at IPL firmly emphasised the importance of other factors in such a programme, including:

- Approval procedures for service providers.
- Critical review and statistical analysis of inspection results.

In the case of HTHA, it is strongly recommended that plant owners develop an inspection programme for HTHA in partnership with their selected inspection provider, and that these relationships be maintained in the long-term, in order to fully optimise plant assets.

Conclusion

The experience of managing HTHA at IPL has highlighted the importance of a robust asset integrity management programme. HTHA cannot be effectively managed in isolation by simple inspection, and a number of factors must be carefully considered in order to maximise the likelihood of detecting and controlling the risk that this damage mechanism presents. Operating such a programme is a continuous activity. It is important to consider the link between risk level and decision making, particularly as it relates to capital expenditure. It is also critical to ensure that all staff appreciate the risk. In order to guarantee this knowledge is gained, it is important that ongoing IOWs, SOPs and training programmes are developed to reflect the risk associated with HTHA.

Inspection for HTHA is an essential part of the asset integrity management programme and it has been found that such inspection is a particularly difficult task requiring significant expertise. Successful detection and quantification of HTHA requires the ability to select the most appropriate technique for any given inspection challenge and optimise that technique. The skill and experience of the inspection personnel is paramount to the success of the inspection, and must be demonstrable to standards embedded in company procedures and practices. **WF**

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4. 'Risk-Based Inspection Technology', API Recommended Practice 581, (April 2016).



500 TPD Nitric Acid Plant, Weatherly



450 TPD Nitric Acid Plant, Hercules



250 TPD Nitric Acid Plant, Chemico

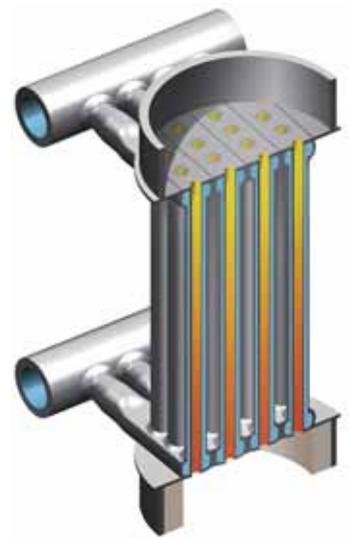
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HTHA Detection and Sizing (HTHA-40)

Our advanced HTHA course is designed to educate Advanced UT inspectors in methodologies and techniques to more reliably detect HTHA. This course is founded on extensive work and experience gained during industrial trials as part of the E2G HTHA JIP. Lavender are custodians of the extensive sample inventory and this will be used for HTHA training and certification purposes. All samples have been validated by metallography and are sourced from pressure retaining equipment retired from service.

Course Content:

The level 2 advanced course will address the following issues;

- The course will contain in depth training on advanced UT applications, fine tuning of equipment parameters and data acquisition from a variety of thickness and states of material degradation.
- Data analysis will consist of many forms of raw ultrasonic data including TOFD, PAUT, TFM and backscatter techniques.
- Velocity ratio, attenuation and frequency analysis will also be addressed.
- Damage mechanisms of HTHA ranging from colonies of microscopic damage through to through wall cracks will be experienced.
- Characterization of material degradation is a critical part of this course. Sources of false positives will be discussed, material inclusions, stepwise HICC damage and weld back cladding issues are also included.

The course will involve hands on testing as well as theoretical approach and offline data analysis tutorials.

Duration:

40 hours held over 5days; end of course assessment exam included.

Pre-requisite:

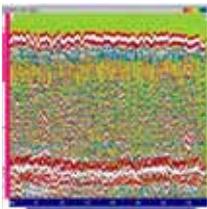
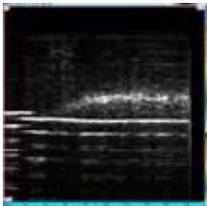
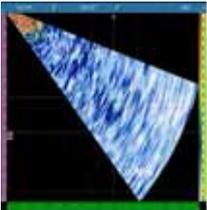
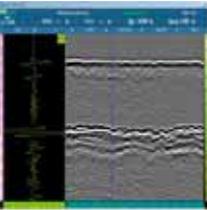
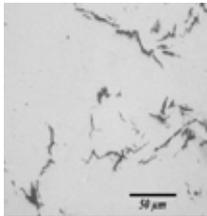
Trainees should ideally hold Level 2 TOFD and PAUT certification.

Schedule:

Courses are scheduled throughout the year. Please contact us for details.

Specific Applications and Support:

Lavender International do not generally perform site inspections. We do however provide company support by assisting in fine tuning inspection set-ups and overseeing data analysis, and where necessary support specialist applications. Please contact Michelle Chapman for more information.



TACKLING TOUGH ENVIRONMENTS

Raquel Rodríguez, Tubacex

Innovation, Spain, outlines a study of steel grades in harsh high temperature and high pressure urea environments.

The materials used for the construction of urea plants often suffer strong corrosion attacks by urea synthesis process fluids (ammonia, water, carbon dioxide, ammonium carbamate, etc.). Corrosion is particularly severe in urea units that operate in high temperature and high pressure conditions, such as the stripper.

The function of a stripper is to remove unconverted ammonium carbamate from the urea solution. As an intermediate compound produced during urea synthesis, ammonium carbamate is characterised by its very intense corrosivity. Furthermore, the working pressure and temperature of this vessel tends to be in the region of 140 – 200 bar and 180 – 210°C respectively. The high temperature, high pressure and presence of ammonium carbamate make the conditions in the urea stripper extremely harsh.

Preventing corrosion in the stripper is key to guarantee the safe and stable operation of urea plants. As a preventive measure, oxygen is usually injected into the process fluid to facilitate passivation on the surfaces of stainless steels. However, this process tends to involve high costs, lower plant efficiency and more safety problems due to the risk of explosive gas mixtures forming. Current trends are aimed at more efficient urea synthesis processes, with higher operating temperatures



Table 1. Chemical compositions of the tested steel grades. Typical values in weight (%)

	S31050	S31803	S32750	S32760	TUBACEX UREMIUM29
Carbon	0.025 maximum	0.03 maximum	0.03 maximum	0.03 maximum	0.03 maximum
Manganese	2 maximum	2 maximum	1.2 maximum	1 maximum	0.8 – 1.5
Phosphorus	0.02 maximum	0.03 maximum	0.035 maximum	0.03 maximum	0.03 maximum
Sulfur	0.015 maximum	0.02 maximum	0.02 maximum	0.01 maximum	0.03 maximum
Silicon	0.4 maximum	1 maximum	0.8 maximum	1 maximum	0.8 maximum
Nickel	20.5 – 23.5	4.5 – 6.5	6 – 8	6 – 8	5.8 – 7.5
Chromium	24 – 26	21 – 23	24 – 26	24 – 26	28 – 30
Molybdenum	1.6 – 2.6	2.5 – 3.5	3 – 5	3 – 4	1.5 – 2.6
Nitrogen	0.09 – 0.15	0.08 – 0.2	0.24 – 0.32	0.2 – 0.3	0.3 – 0.4
Copper	-	-	0.5 maximum	0.5 – 1	0.8 maximum
Other	-	-	-	W 0.5 - 1	-

Table 2. Corrosion rate and maximum depth of attack after autoclave immersion tests

	S31050	S31803	S32750	S32760	TUBACEX UREMIUM29
Corrosion rate (mm/yr)	1.2510	0.6097	0.3380	0.4177	0.1331
Maximum depth of attack (µm)	40	40	25	35	15



Figure 1. Stripper tubes produced by Tubacex.

and pressures and a drastic reduction of injected oxygen, making the development of more suitable robust materials necessary.

Tubacex currently manufactures tubes and pipes of several corrosion resistant materials for use in urea plants, as well as specifically for urea strippers. An

example of manufactured stripper tubes is presented in Figure 1.

Many different corrosion mechanisms can be used in urea environments. The experiments outlined in this article were developed to reproduce the very corrosive conditions of a urea stripper but without oxygen injection, making the environment inside the experimental setup more harsh than a real on-stream situation. The main objectives of this study were to identify the basic mechanisms controlling the corrosion of different stainless steels in the urea synthesis process and to establish the relationship of these mechanisms with the chemical composition and the microstructure of the tested steel grades.

Autoclave immersion tests

The corrosion of different stainless steels in high temperature and high pressure urea environments has been analysed by means of autoclave immersion tests to investigate the corrosion mechanism in each case. A chromium-high (Cr-high) nickel-high (Ni-high)-molybdenum (Ni-Mo) austenitic stainless steel that is commonly used in urea equipment and several duplex type stainless steels were involved in this study. The results showed that UNS S31050 experiences active corrosion due to the absence of oxygen in the experimental setup, whereas TUBACEX UREMIUM29, a superduplex stainless steel that was developed specifically for use in urea plants, remains passive and with the lowest corrosion rate.

To specify, the five materials investigated in this work were as follows:

- UNS S31050: 25Cr-22Ni-2Mo austenitic stainless steel.
- UNS S31803: 22Cr-5Ni-3Mo duplex stainless steel.
- UNS S32750: 25Cr-7Ni-4Mo superduplex stainless steel.
- UNS S32760: 25Cr-7Ni-3.5Mo-0.75W superduplex stainless steel.
- UNS S32906: 29Cr-7Ni-2Mo superduplex stainless steel.

Each of these steel grades can be applied in a different section of a urea plant, and are expected to have good corrosion resistance in the urea environment. More detailed chemical compositions are shown in Table 1.

The specimens used in the experiments were machined from industrially-produced solution annealed tubes and pipes. The immersion tests were conducted in a 5 l zirconium autoclave, which had been designed

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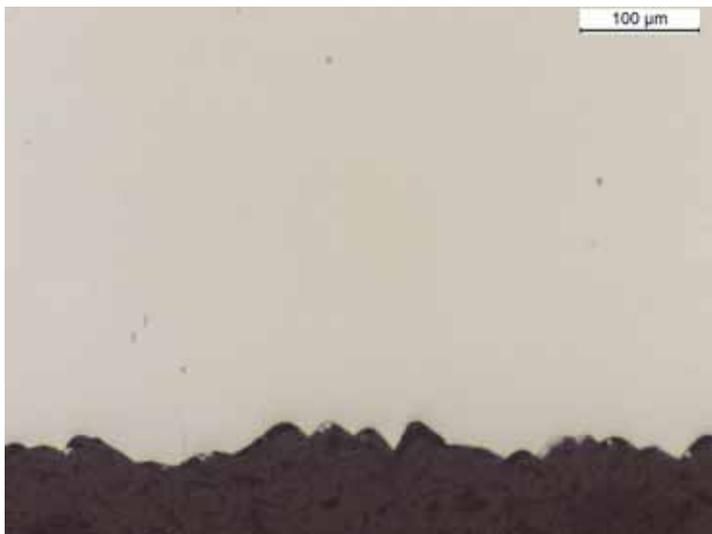


Figure 2. Cross section of the S31050 test specimen after a 15 day immersion in the autoclave.



Figure 3. Cross section of the S31803 test specimen after a 15 day immersion in the autoclave.

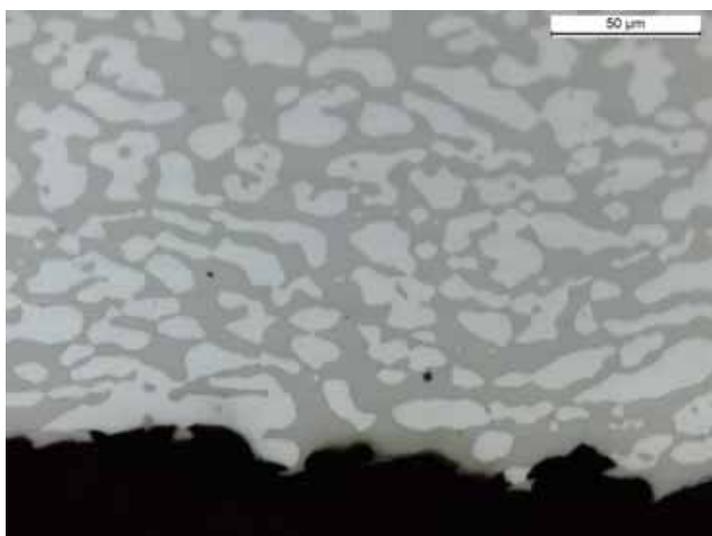


Figure 4. Cross section of the S32750 test specimen after a 15 day immersion in the autoclave.

specifically for these tests. The autoclave was equipped with adequate feed, discharge lines and a stirrer. The test solution contained a mixture of urea, ammonia and water, at concentrations that are characteristic of the urea synthesis process. The temperature and pressure of the experiments were set in the upper level of the typical ranges that are measured in a urea stripper, 180 – 210°C and 140 – 200 bar. The test solution was deaerated before starting the tests to remove oxygen from the system.

The experiments were designed to simulate the most severe conditions achievable in the stripper of a urea plant without oxygen injection. Test specimen preparation followed the ASTM G31 (Standard Practice for Laboratory Immersion Corrosion Testing of Metals) standard indications and corrosion rate was measurement using the gravimetric method.

The testing procedure took 15 days. Once the tests had been finalised, sections of the samples were prepared and observed by optical microscopy to analyse the type of attack experienced in detail.

Corrosion rate and corrosion mechanisms

The corrosion rate and measured depth of attack for each steel are summarised in Table 2. The maximum depth of attack was calculated on the surface of the remaining tube wall thickness.

As can be observed in Table 2, the corrosion rates of the analysed materials in descending order are as follows: S31050, S31803, S32760, S32750, TUBACEX UREMIUM29.

TUBACEX UREMIUM29, a superduplex with very high Cr content (approximately 29%), was specially developed for application in the urea industry.

Figures 2 – 6 depict cross sections of the five test specimens after 15 days of immersion in the autoclave. Considering the measured corrosion rates and the metallurgical investigation on the test specimens, the following aspects can be highlighted for each steel grade:

UNS S31050

With the highest corrosion rate, UNS S31050 experienced a uniform attack. This high corrosion rate is related to the absence of oxygen in the autoclave, which prevents stable passivation. This austenitic grade steel is an established, successful material for use in urea environments, including urea strippers, as its corrosion rate when oxygen is present is low.

UNS S31803

The microstructure of UNS S31803 after chemical etching reveals no preferential attack on the ferrite or austenite phase. Under the test conditions, both phases suffered some corrosion, and the corrosion rate was quite high. The reason that this duplex stainless steel behaves this way is the combination

of a lack of oxygen in the experimental setup and insufficient chromium content in the matrix.

UNS S32750

Similar to UNS S31803, UNS S32750 experienced a generalised attack of both the austenite and ferrite phases. The corrosion rate of this superduplex grade is lower than UNS S31803 due to higher chromium and molybdenum contents in the matrix. The measured corrosion rate and type of attack indicated that the chromium content of this steel is not enough to guarantee passivation in the conditions reached in the autoclave.

UNS S32760

UNS S32760 showed similar behaviour to UNS S32750, with an overall attack through both the austenite and ferrite phases, but had a slightly higher corrosion rate. This fact is possibly related to the lower Mo content in the UNS S32760 matrix, as this element is partially substituted by tungsten in this grade. Tungsten can be less effective for corrosion resistance in the conditions achieved in the autoclave. The chromium content of this grade is not considered sufficient for it to be used under the harsh conditions of a urea stripper.

TUBACEX UREMIUM29

Selective attack of the austenite phase were detected on this superduplex grade during the testing. TUBACEX UREMIUM29 showed the lowest corrosion rate with the lowest depth of attack. Its chromium content was high enough to guarantee the passivity of ferrite phase under the conditions inside the autoclave. The austenite phase suffered a slight attack as no oxygen was present during the test. This preferential attack of the austenite has been reported to disappear at longer exposure times, as anodic protection of the austenite phase can occur when the ferrite/austenite area has become large enough as a result of corrosion of austenite. Even lower corrosion rates are then expected as the time in service increases.

In this study, where the conditions in the autoclave were extremely harsh (high temperature, high pressure, corrosive test solution, and an absence of oxygen), this grade showed high corrosion resistance.

Conclusion

In the preceding study, autoclave immersion tests were designed to simulate the highly corrosive conditions of a urea stripper. Oxygen was eliminated from the experimental setup due to the current tendency of decreasing the amount of oxygen that is injected in order to reduce costs, as well as increase plant efficiency and safety. Thus, the atmosphere generated inside the autoclave was more severe than that present in real strippers.

During the testing of the five different steel grades, high Cr-high Ni-Mo UNS S31050 austenitic stainless steel



Figure 5. Cross section of the S32760 test specimen after a 15 day immersion in the autoclave.

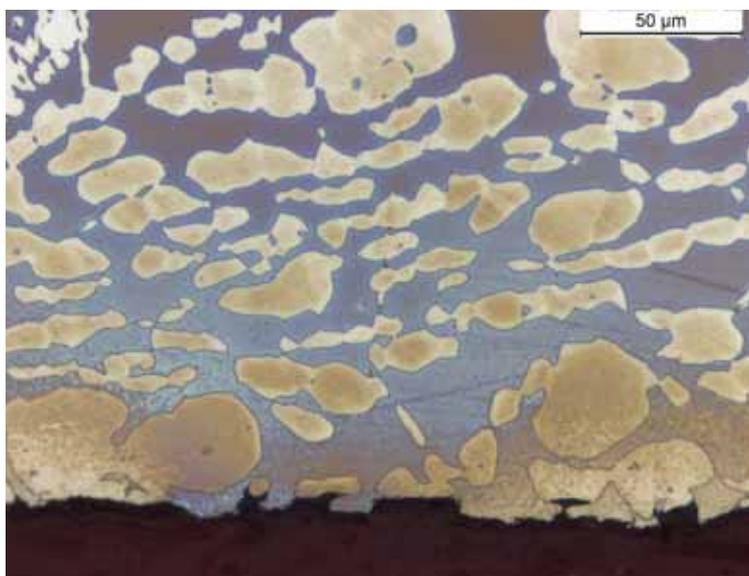


Figure 6. Cross section of the TUBACEX UREMIUM29 test specimen after a 15 day immersion in the autoclave.

experienced the highest corrosion rate with a generalised attack as austenite cannot be passivated under the above-mentioned conditions. However, this grade would have shown a very low corrosion rate if some oxygen was present. Duplex type stainless steels, such as UNS S31803, UNS S32750 and UNS S32760, showed medium corrosion rates and a uniform attack of austenite and ferrite phase. This occurred because the chromium content of these grades is not high enough to reach total passivity under the test conditions. TUBACEX UREMIUM29 (UNS 32906), a superduplex with the highest chromium content and a chemical composition, developed specially for urea environment, showed the lowest corrosion rate and only a slight selective attack on the austenite phase due to the absence of oxygen. **WF**

PRACTICE — M A K E S — PERFECT

**Dieter Krenz, Linde, Germany,
Manel Serra, Inprocess, Spain, and
Theron Strange, Simplot Phosphates,
USA,** discuss dynamic process simulation
in an ammonia plant startup.

Process simulation is a technology used in many areas and project phases. Its usage ranges from the design stage of a plant (initial concept and front-end engineering [FEED]) to assistance in daily plant operations, including planning, optimisation and monitoring. To a certain extent, the simulation lifecycle is similar to the lifecycle of a process plant, bringing support and value to multiple stages in the lifetime of a process plant.

Using Simplot Phosphates' ammonia process plant in Rock Springs, Wyoming, US, where dynamic process simulation has been used, as an example, this article focuses on the implementation and benefits of an operator training system (OTS) for the plant. It will explain this specific use of the OTS, and will outline some experiences and results from the point of view of the three parties involved in the project:



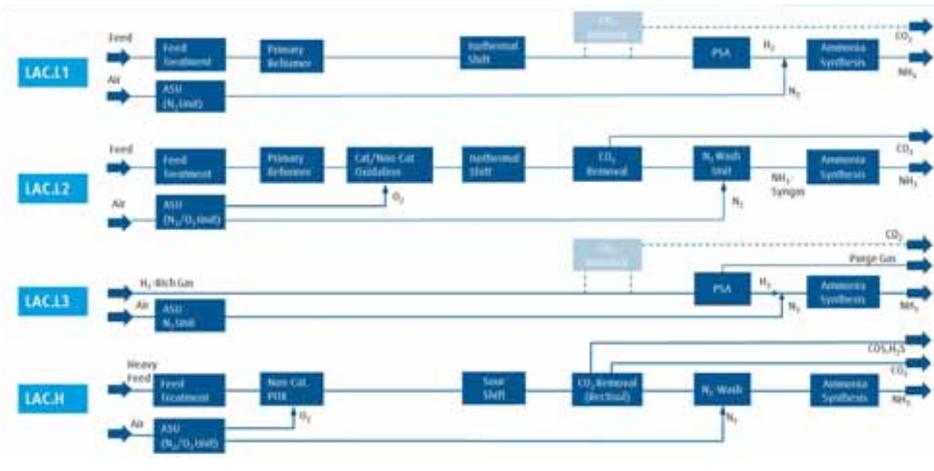


Figure 1. Linde's four ammonia concepts.

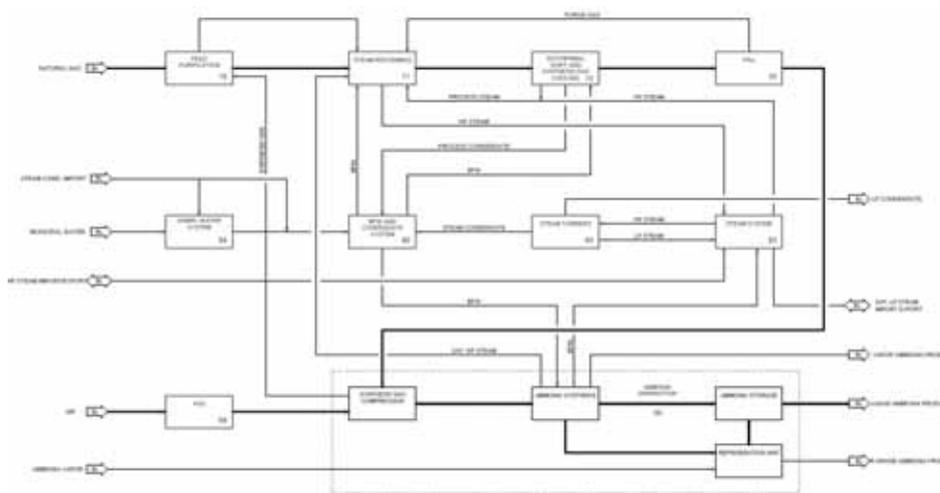


Figure 2. A block diagram of the simulated ammonia plant.

the licensor and engineering company in charge of the construction, Linde; the plant owner and operator, Simplot Phosphates; and the simulation services provider in charge of developing and deploying the OTS, Inprocess Technology and Consulting Group, S.L.

Ammonia plant training

After Simplot Phosphates decided to construct a new ammonia plant to supply its Rock Springs site, the company also saw the need to provide appropriate training to those who would eventually operate the plant. There were several reasons for looking carefully into training, including that the ammonia plant would introduce a new process that the current site operators were not familiar with and there would be a significant number of people hired to operate the plant with limited experience in the environment.

In addition, because Simplot Phosphates sought high operational performance, it also wanted to both gain an in-depth understanding of the process and push the limits of its plants to increase efficiency and throughput. Quality training and excellence in operation crystallised into the OTS project that was co-ordinated and supported by Linde, delivered by Inprocess, and powered by a rigorous dynamic simulation engine. Operator training using the system was delivered by Linde.

The scope of the OTS

To provide some context to this case, its scope involved a new Simplot Phosphates ammonia plant project in Rock Springs that was designed according to Linde's LAC.L1 concept, using a natural gas feedstock. This is one of four of Linde's ammonia concepts, all of which are shown in Figure 1. With the LAC.L1 concept:

- An inert-free ammonia synthesis gas in the right H_2 to N_2 ratio is generated.
- A steam reforming based H_2 plant with an isothermal shift reactor is used.
- Pure hydrogen and pure nitrogen (from an air separation unit) are directly available from the process streams.
- The ammonia plant remains energy efficient.

The basic LAC.L1 diagram in Figure 1 became the actual design used at the Rock Springs plant, as shown in Figure 2. With the exception of the demin water system, all of the major units are part of the scope of the OTS and were modelled in the dynamic process simulator.

Simulation lifecycle

The primary objective of an OTS is to provide a plant specific simulation environment for both initial and refresher training of those who operate the control room or other plant equipment operators. Hence, as well as the importance of the distributed control system (DCS) and safety instrumented system (SIS) emulation, another key point to consider is the simulation model that will support the OTS. The virtual plant must be high in fidelity and have a wide scope for better training. It must also run quickly, be robust and support a range of operating conditions, from cold startup up to maximum throughput.

High fidelity, customised dynamic process models built around software such as AspenTech's HYSYS®, Honeywell's UniSim® or VMG's VMGSim® and derived from the detailed design of the real plant process units, are powerful predictive tools. They can extrapolate process conditions far from nominal conditions, which extends the benefits that an OTS can provide beyond the training objectives. Figure 3 summarises this extended OTS usage.

The OTS model can be re-used for holistic evaluation of project functionality throughout an entire project. The simulation project will, in turn, benefit different project phases, including concept, FEED (pre-engineering), detail engineering, commissioning, startup, and operation of the plant. The complexity of the simulation model increases

during the execution of a project in order to fulfil specific engineering requirements. The right level of detail is needed to predict realistic behaviour without having to collect and use vast amounts of plant and design data.

Using the same process model as a base for the engineering studies, along with operational support and operator training systems, reduces the cost of developing and maintaining tools. It also maximises return on the OTS investment.

While lifecycle simulation projects are clearly suited to grassroots plants, brownfield plants can still experience the benefits of using high fidelity dynamic simulation models.

If developed and implemented correctly, the use of high fidelity models throughout a plant's lifecycle will contribute to significant learning time reduction and greatly increase operators' skills in terms of control. They can also substantially boost confidence in operating procedures while offering the possibility of testing new strategies for optimal, safe and energy-efficient operation, without influencing the actual operation.

OTS and dynamic simulation models

The hydrocarbon process industries have used OTSs with a key focus on accident prevention since the mid-1970s. An OTS is often viewed as an important, if not critical, training tool for grassroots plants and process units that are being revamped or re-instrumented.

OTSs are suitable platforms to practice on because they provide an accurate representation of the plant, which allows trainees to perform unfamiliar process and control tasks in a safe environment. Any mistake or error can be converted into a lesson learnt without any real life consequences. It is key to keep practicing the procedures regularly, particularly when a modification is implemented in the plant or control/safety system.

When thinking of an OTS, the usual expectation is that the system delivers an accurate replica, containing the following components of the processing plants:

- The process, represented by individual pieces of equipment (such as columns, compressors, heat exchangers and their connections).
- Field instrumentation (such as manual valves, level gauges).
- DCS or programmable logic controllers (PLC) for governing the plant and its equipment.
- Sequential logic and emergency shutdown (ESD) systems.
- Control room operator consoles.

The current trend in operator training simulators is to connect a dynamic simulation model (Figure 4) to a copy of the actual plant DCS and a copy of the human machine interface (HMI). Depending on how these copies are developed and implemented, different commercial solutions are offered.

The DCS can be reproduced in the same dynamic simulation model, with the controllers available in the process simulation platform. Alternatively, a DCS emulation software can be used for these purposes.

Similarly, the operator console HMI can be emulated by a specific graphical and programmable software. An



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3 STAGE BENEFITS FROM OTS

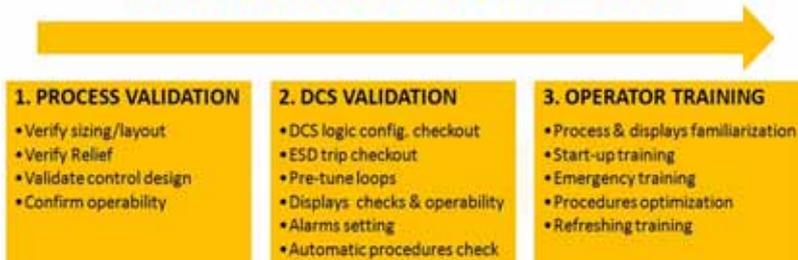


Figure 3. A simulation lifecycle and the benefits of the OTS.

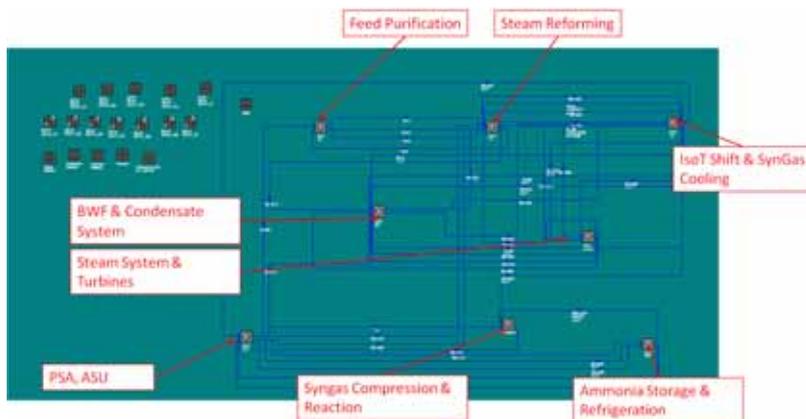


Figure 4. A dynamic simulation model.

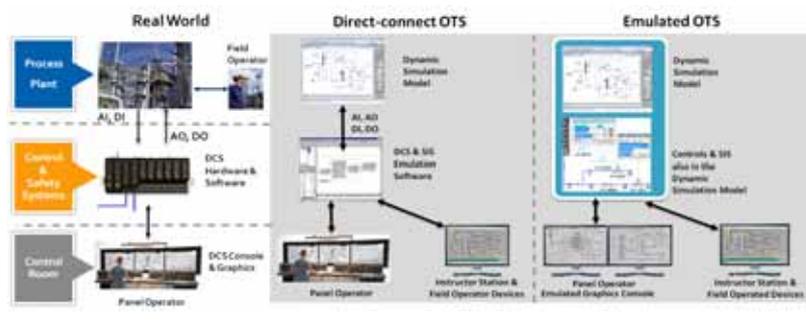


Figure 5. The OTS types and elements compared to a real plant.

alternative is to replicate the operator console using the DCS HMI software or in DCS vendor consoles.

The OTS for Rock Springs involves a stimulated or direct connect OTS, as shown in Figure 5.

A direct connect option is characterised by the fact that dynamic process models and controls are separate applications. Dynamic process models are generally simulated by a high fidelity process simulator, while the controls (including ESD, FGS and PLCs) are simulated through proprietary DCS vendor software (and/or hardware, if required), reproducing the actual DCS controllers' behaviour. Hence, the DCS-implemented logic for the simulator and the plant is the same.

Consequently, standard OTS architecture allows for communication between the replica DCS controller computer(s) and the process models computer(s), before sending the signal to the user (instructor or operator) on their screens, which, for a direct-connect OTS, are also an exact replica of the actual plant's control panels.

Although a stimulated solution may have a greater initial investment, it is an effective solution in terms of its fidelity to the plant's actual control and safety, and in terms of ease of maintenance. Also, the dependency of this type of OTS on the availability of the DCS database requires careful management of schedules and a flexible project execution methodology (supporting concepts such as a staggered delivery approach) in greenfield projects.

The OTS virtually emulates the plant in detail. This includes all of the DCS points, interlock logic, process controls and overall plant operation, such that the trainee sees minimal differences between the simulator and the actual plant. All operator-related activities that are performed in the real life main control room can be conducted using the OTS.

The trainee uses the plant user interface to control the simulated process via the stimulated DCS. The instructor can set malfunctions and other controls in the simulation, while accessing the trainee's station screen to see their actions. The instructor also tends to undertake field actions, as requested by the operator.

Uses and benefits of an OTS

As mentioned previously, OTSs are primarily used to train control room operators on various aspects of plants. An OTS can give trainees the equivalent of about six years of on-the-job training. Periodic training using the system, especially on critical emergency scenarios, brings operator skill levels as high as possible. Real time dynamic allows trainees to experience every type of operating condition in a 'hands-on manner', virtually. Types of scenarios include: normal operation, unusual operating scenarios, startup from cold conditions, restart from tripped conditions, shutdowns, equipment malfunctions, emergency conditions and process upsets, as well as communication with field operators.

emergency conditions and process upsets, as well as communication with field operators.

Instructors have tools to develop standard exercises for testing and evaluating each student's performance, as well as documenting the results of the training session. Training sessions can be repeated to monitor and prove trainee improvements.

Training using an OTS is cost-effective as it allows trainees to experience more operating situations in a relatively short period of time, while also under supervision. This helps reduce personnel errors and maximise plant availability, in turn increasing overall safety with reduced risk of expensive equipment damage. Using an OTS also provides knowledge of dynamic processes, which allows for anticipation of the unexpected.

Although the primary use of OTSs is operator training, they also allow control strategy tests to be performed, and operating procedures to be developed, verified and compared.

It can also debug and verify DCS configuration in a dynamic 'near plant' environment, which results in less interrupted commissioning. With these uses in mind, the following benefits can be achieved using an OTS:

- Minimisation of startup time and achieving steady production more rapidly.
- Achievement of an easier, stable and ongoing production.
- Reduction or prevention of operating incidents, unnecessary trips and emergencies, which leads to an increased on-stream production rate and a reduced risk of equipment damage.
- Increased operator confidence, which allows plants to be operated closer to product specifications and process constraints.

OTS use in Rock Springs

In the Rock Springs case, the OTS was delivered before the plant's startup to accommodate the training requirements with the availability of the DCS database. This allowed the operator training to be undertaken in time, following a pace to match that of the startup of the different areas of the plant.

During a three month training programme, Simplot Phosphates' trainees were familiarised with the plant's startup procedures, DCS functionality (HMI, control logic, etc.) and how to communicate with field operators.

Although many trainees were not familiar with a DCS and the ammonia plant process, the training gave them sufficient

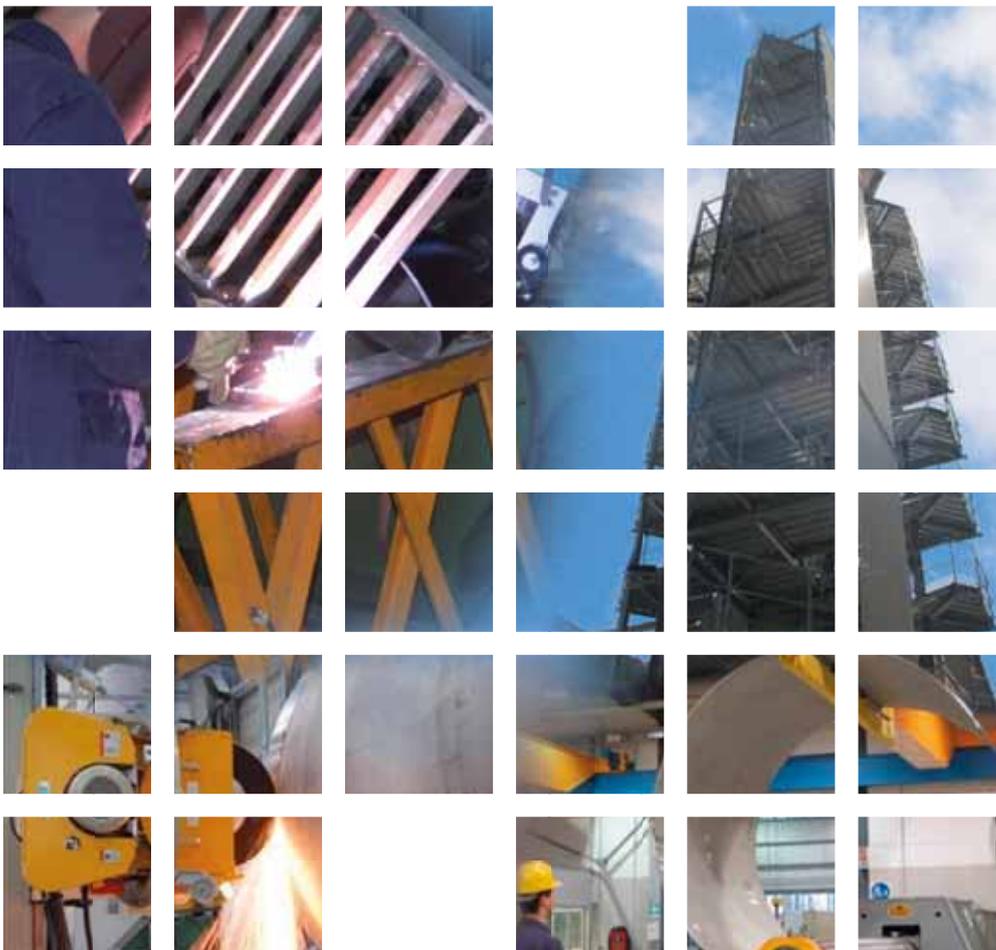


Figure 6. Simplot Phosphates' Rock Springs OTS training room during the OTS commissioning.

confidence to startup the plant and recognise unstable conditions early on. This led to faster and more effective response times, as well as less lost time.

When implementing the OTS, bugs in the DCS database were detected and eliminated. Running the startup procedure allowed for an extensive evaluation of written procedures, pointing to the need for clarifications, and the potential for changes and optimisation, which were later realised during the actual commissioning of the plant.

OTSs give operators refreshment on the specifics of the plant, with the simulator being continuously used to maintain and test operators' skills. **WF**



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SIMULATION FOR SAFETY

Todd Jaco, Emerson, USA, explains how using an effective ammonia plant operator training system can greatly reduce startup time.

One of the larger producers of nitric acid for use in fertilizers in the US uses ammonia (NH_3) as a key component in its operations. This company faced an issue: it had been purchasing ammonia from a pipeline but decided to build its own ammonia plant to gain a significant cost advantage. While fertilizers consume 80% of the total world production of ammonia, it can also be used in the production of fibres, plastics and pharmaceuticals, to name a few.

As supplies can be tight with so many operations making use of it, being able to produce your own and sell the excess back to the market is largely beneficial for fertilizer producers.

The company not only had to work through the design, procurement and construction of its new ammonia plant but also needed to find skilled workers to operate its facility. Its location is remote enough that it is difficult to find a large number of well-trained, experienced operators. In addition, the

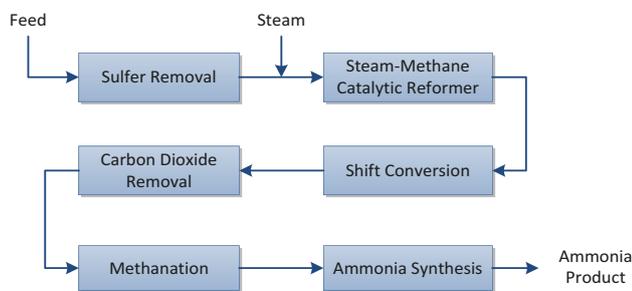


Figure 1. A block flow diagram of the ammonia production process. Source: Milton Beychok.

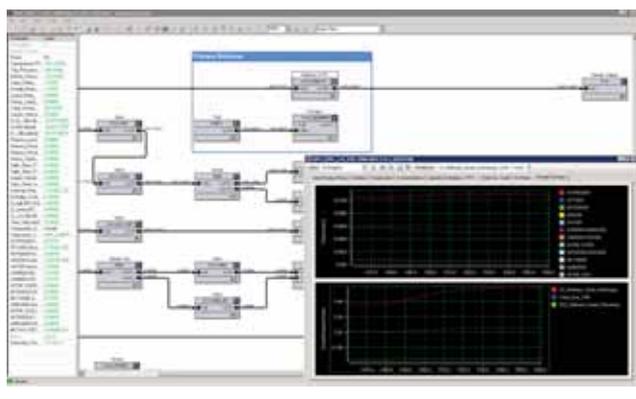


Figure 2. The Mimic modelling environment, Simulation Studio, following IEC 61131-3.

plant faced hiring pressure from other industries vying to hire the best of the limited pool of experienced workers. Thanks to advanced simulation technology, the company was able to train operators in startup and shutdown, as well as how to handle rare process excursions that they may have not seen with even 10 years of experience. All of this took place before the physical plant was built and operational.

Running an advanced simulator can be extremely similar to running an actual ammonia plant. Not only are the trainee operator stations at the simulator identical to what they will see when the plant starts up, but the feel of running the equipment is very realistic. Simulation and the supporting models are a way to capture the expertise of skilled personnel. Initial and future training will instill this knowledge in operators, with the result being smoother and more efficient plant operations.

The challenges of ammonia production

Starting an ammonia production plant from scratch can be a risky business. Making ammonia involves reacting one part nitrogen with three parts hydrogen at temperatures of 400 – 450°C and at pressures of up to 180 atmospheres. The operating pressure and temperature are a compromise between the need to produce ammonia and the cost of running pumps, as well as the added capital expense that arises from using suitably strong pipes and reaction vessels.

In making ammonia, a hydrocarbon (such as natural gas) is processed to produce hydrogen while atmospheric nitrogen is the source for the other feedstock. Because the nitrogen-nitrogen bond is strong, iron-based catalysts are

used to prod the reaction along. The single pass ammonia yield is only approximately 15%, but using multiple passes boosts the yield to above 95%.

The block diagram in Figure 1 illustrates this multi-step process. It requires careful control due to the presence of hydrogen sulfide, hydrogen and carbon monoxide during processing. These gases are poisonous, corrosive, flammable and explosive. Hence, manufacturing ammonia can require extensive capital investment to ensure proper process control and safety.

The production process includes primary and secondary natural gas reformers that free the hydrogen. There also are shift reactors to convert carbon monoxide to carbon dioxide. Sulfur is removed from the feed at the beginning of the process because it kills the catalysts and hydrogen sulfide (H₂S) is eliminated by reacting it with zinc oxide. The final step before ammonia synthesis removes small residual amounts of carbon monoxide or dioxide from the hydrogen stream.

A solution

At first glance, it might seem that the ideal solution for training operators for ammonia production would be to have them practice startup, shutdown and responses to various scenarios using actual equipment. However, taking such an approach in the real world is not practical or efficient.

Most importantly, training with real equipment would be expensive and dangerous. There is a specific sequence that must be followed to bring boilers and other process modules online that must be followed exactly. In addition, high pressures must be dealt with, not to mention dangerous gases. The correct sequence of actions is specified in the plant's standard operating procedures. The same documents also contain instructions about how to handle common process problems, including stuck valves, pump failures and boiler mishaps.

If actual plant equipment is used, failure by operators to follow recommended procedures could result in damage to expensive equipment, harm to plant personnel or even cause an event that is noteworthy enough to make the national news. What is more, deliberately putting a plant in an unsafe operating condition as part of a training exercise could result in regulatory review or litigation, if and when the practice came to light, even if nothing catastrophic happened.

Turning to the case introduced earlier, though the experience pool was limited in the area, there were some seasoned industry veterans that the new plant could draw upon. With decades of experience, these professionals are capable of standing on the plant floor, listening to the machinery and determining, despite the din, whether a particular pump is having problems and is in need of replacement soon. However, their long work history also means many are nearing retirement and will soon leave, taking their expertise with them. An efficient solution, therefore, would codify this accumulated knowledge and make it available for use by others.

Training with a live plant is not the best way to transfer expertise from more to less experienced personnel. Working in an actual plant would not ensure the capture

of knowledge because rare process upsets do not, generally, occur very often. In fact, the client's new plant may not experience the upsets before many of the experienced employees retire.

The virtual plant

Confronted by the need for safe and effective training, as well as the requirement to capture scarce expertise, the company decided that dynamic simulation was the right solution. This approach enabled trainees to run through startup and shutdown procedures with no risk to anyone. Those using the simulator can gain valuable experience and the training can take place both before and during plant construction. Also, a parallel training and construction approach minimises delay in plant startup.

In addition, using the simulator can allow scenarios such as pump failure, valves sticking open or closed, pipe ruptures or other rare events to be devised. Familiarity with such process excursions could be critical to the ability of operators to successfully resolve them, should they occur.

This approach presented the company with its own set of requirements. Chief among them was that the simulation had to be lifelike. That is, there has to be an extremely high degree of similarity between the simulation and the actual plant. Otherwise, the training would be counterproductive, with operators familiarising themselves with something that diverged from the reality they would face.

Beyond training

Dynamic simulation offers a number of benefits beyond operator training. It can aid in the capture of the knowledge and the lessons learnt by experienced operators, thereby ensuring that expertise is not lost. It can also help in the analysis of both safety and environmental risks. Dynamic simulation can aid in process optimisation by allowing process improvement studies. During the capital phase of a project, it can also assist in testing control systems for operational readiness by allowing lifelike exercising of the distributed control system. This testing can include both the basic process control (BPC) and safety instrument systems (SIS). There are also a number of other areas in the capital and operational phase of a project that could benefit from dynamic simulation.

In addition to high fidelity, other important simulator characteristics are its ability to communicate with external control systems and its use of modelling standards. The first ensures that control systems can be fully exercised, while the second avoids problems that can arise from proprietary solutions.

Simulating and ammonia

After considering the alternatives, the company in this case selected Mimic™ simulation software from



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Figure 3. Simulation allows small teams to fill in where large teams were once needed.



Figure 4. Virtualisation greatly reduces the hardware footprint of complex simulation systems.

MYNAH Technologies. As of 19 May 2017, MYNAH Technologies was acquired by Emerson. Among other things, the simulator has an ammonia plant module with OPC server access to all model parameters. The simulator also offers direct IO simulation for most modern control systems. All model objects within the simulator are industry standard IEC 1131 function blocks, as shown in Figure 2. For ammonia production, the dynamic simulator includes replicas of the following important process areas:

- A natural gas primary reformer, coupled with a waste heat boiler.
- A natural gas secondary reformer.
- High and low temperature shift reactors.
- An acid gas removal system.
- A methanator.
- An ammonia converter.
- Auxiliary separators, heat exchangers, fluid movers and piping.

Each of these areas is rigorously modelled, ensuring that the entire process can be simulated with a high degree of accuracy. Doing so involves dynamic real-time mass and energy balances, vapour-liquid equilibrium, catalysed reforming and oxidation reactions, along with other factors.

Once its selection process was complete, the company approached MYNAH with a desired schedule. While it is

not uncommon for complex ammonia plant simulations to require nearly two years to develop, the company required the simulator within a 16 month window. During that span, a design specification would have to be devised and approved, followed by development of the simulator. The timeline ran from August 2014 to December 2015.

The control system for the plant was Rockwell Automation's PlantPAX®. The simulation uses the actual plant control system configuration running in a virtual environment so there is no need for the actual control system hardware to be deployed for the simulator. Because it offers integrated simulation of BPC and SIS, both basic process control and safety instrumented system modules can be designed and tested. Mimic simulation software communicates directly with the control logic through an API that was developed by MYNAH. The control system believes that it is communicating with real equipment in the field, providing a highly realistic experience to the user.

By early October 2015, MYNAH had fully developed the simulator for training new operators. There were some minor updates that were required to finalise the delivery, but the 60 plus operators were able to begin training on the system. Though it was not part of the overall plan, the engineering team was also able to use the simulator to validate and test the turbine and combustion control systems, speeding up the process of finalising those systems.

Results

The simulator underwent FAT in late 4Q15, several months before the projected completion of the plant and its expected startup. Though there were mechanical setbacks that impacted project completion, thanks to the use of the simulator in operator training, the startup from an operations standpoint was extremely effective and ran smoothly.

Conclusion

The simulator has and will continue to positively impact operator training. Current employees will be well versed in how to start up and shut down plants, with the high reliability of the simulation ensuring that these training exercises are effectively indistinguishable from the real thing. Operators will have experience in how to handle process excursions, with the result being that they will be better prepared than they otherwise would have been for these unusual occurrences.

Importantly, the development of training scenarios is ongoing. As new ones are devised, these will capture the expertise and knowledge of more skilled operators, which will then be used to train new operators and develop the plant's staff.

The result of this successful simulation has been a more efficient and smoother operating plant, with the added benefit of a faster ramp to full production due to a well-prepared operations team. This assured that the company's ammonia plant will be able to meet the production demands of the downstream process units at the site, solving the problem that the plant was built to tackle. **WF**

INCREASED

P₂O₅

Curtis Griffin, PegasusTSI, USA, and Shannon Griffin, University of South Florida, Chemical Engineering, USA, detail process technology and pilot plant results for the recovery of P_2O_5 from phosphate mine tailings and clays.

The phosphate ore beneficiation process produces two reject streams called 'clays (slimes) and tailings'. Both the clays and tailings can contain a significant amount of P_2O_5 , depending on how well the beneficiation plants are operating. Operating conditions, such as changes in the physical properties of phosphate rock, production rates and ore particle size, can have a significant impact on the amount of P_2O_5 in the clays and tailings. The clays and tailings are typically pumped to settling areas for storage and the P_2O_5 in both reject streams is typically lost. Recovering the P_2O_5 in the clays and tailings would result in increased revenue and mine life.

The study discussed in the following includes pilot plant test runs that evaluated the recovery of P_2O_5 from phosphate clays and tailings.

The pilot testing for the recovery of P_2O_5 from clays was performed as a combined effort with

PegasusTSI and EcoPhos. The phosphate clays were tested with the EcoPhos technology in a preliminary evaluation phase. The objective was to demonstrate the feasibility of using phosphate clays to produce dicalcium phosphate (DCP).

The pilot testing for the recovery of P_2O_5 from tailings was performed as a combined effort with PegasusTSI and Florida Industrial and Phosphate Research Institute (FIPR). Various flotation runs were conducted using different reagents. The results

55

EFFICIENCY

from each run included reagent dose rate, P_2O_5 grade into the flotation cell, P_2O_5 grade exiting the flotation cell and percent recovery. The performance of each reagent as a function of dosage was also monitored.

Florida phosphate mining

Phosphate mining in Florida dates back to the first hard rock deposits found near Hawthorne in Alachua County in 1883. In the early years, phosphate mining was done with wheelbarrows, picks, shovels and mule-drawn scrapers. Mechanised excavation began between 1900 and 1905 with steam shovels. Early steam shovels held only 1 yd³ of earth, but one steam shovel operated by three men did the work



Figure 1. A 1930 flotation cell with wooden launder and floors.

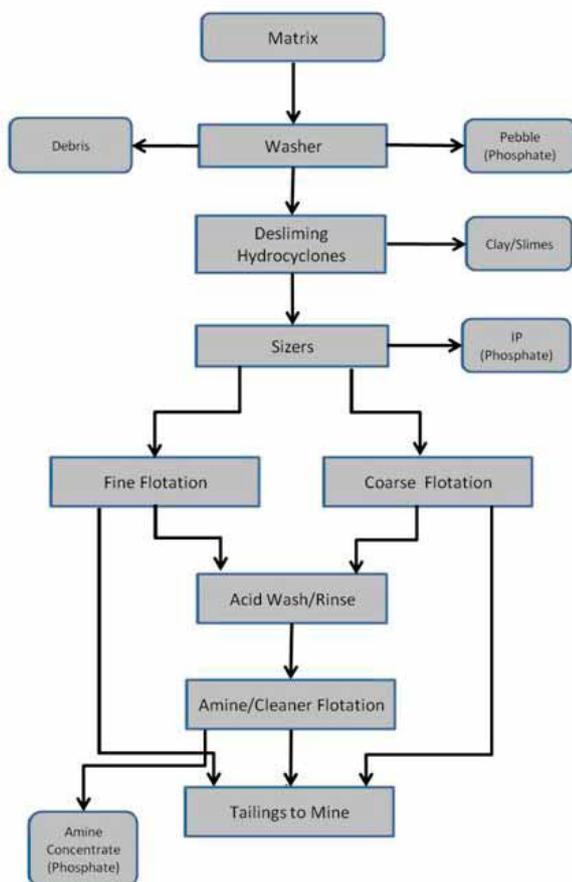


Figure 2. Typical phosphate plant flowsheet.

that 80 men can do by hand. Steam dredges and barges came into use in hard rock areas where the water level was too high for picks and shovels. Centrifugal pumps mounted on barges were also used to mine the river-pebble phosphate deposits in the Peace River until river-pebble mining ended in 1908.

Draglines, the current mining tool, came into use with the dawn of electricity and diesel power in the 1920s and 1930s. By 1930, these electrically driven draglines were adopted as the most economical way to mine land pebble. The following is a basic timeline of the beneficiation evolution:

- 1906: Original flotation patent.
- 1925: Laboratory flotation of phosphate.
- 1927: First flotation pilot plant.
- 1931: High solids conditioning.
- 1935: Tall oil was used as flotation reagent.
- 1939: Sized flotation feed.
- 1949: Hydro-cyclones for de-sliming.
- 1976: Prototype column cell installed.
- 2002: Eriez float column introduced to phosphate industry (Figure 1).

Beneficiation

Beneficiation is the second step in the mining process, after removal of the ore from the ground. It is the technical term describing the industrial process of mechanically separating minerals from each other. The phosphate ore, or 'matrix' as it is called locally, contains three different mineral groups, which must be separated from each other:

- Phosphate minerals.
- Clay minerals.
- Quartz or sand.

Two steps of the beneficiation process result in loss of P_2O_5 :

1. De-sliming with hydro-cyclones.
2. Flotation process as tailings (Figure 2).

Clays (slimes) generation

As mined, the phosphate and sand particles are embedded in compacted mud, or 'clay-balls'. Before separation can begin, all of the particles must be liberated from the matrix of mud. The very first unit operation in the beneficiation process is to disaggregate the various particles; this actually starts while the matrix is flowing through the multi-mile pipeline from the mine to the beneficiation plant. While in the pipeline, the matrix is exposed to shear forces as it passes through the various centrifugal pumps along the pipeline. These intense shear forces cause a significant percentage of the sand and phosphate particles to be liberated from the clay-balls by the time they arrive at the plant.

Once reaching the plant, the first goal is to finish disaggregating the clay, and follow that by making a size separation typically at 1 mm. This processing is conducted in the washer. In all of the currently operating beneficiation plants, the washer is a large structure that receives the matrix, screens it and then discharges a +1 mm 'pebble' phosphate product and a -1 mm slurry of liberated clay, sand and phosphate particles.

The next process objective is to remove the clays, which are finer than 0.1 mm. To reject the clay, all that is required is to size at 0.1 mm and discard the fine fraction. The beneficiation plant does this with hydro-cyclones. Slurry from the washer is fed tangentially into the cyclone (a conical chamber) at a high G force. The slurry swirls around inside the cyclone until fines overflow the top of the chamber. Coarse sand and phosphate particles swirl to the bottom of the cyclone and exit. The fine clays are collected and pumped to large ponds, a significant amount of P_2O_5 can be lost as clays. The clays can be as high as 33% of the total matrix feed and can contain as high as 15% P_2O_5 in some cases, typically a much lower P_2O_5 content is seen (around 2 – 9% P_2O_5).

Recovery of P_2O_5 from clays is very difficult due to the ultra-fine particle size and colloidal particle, typically 50% below 1 μm .

Previous research

Hazen Research was issued a patent in 1969 on a process to leach the clays with sulfuric acid, then to recover the $\%P_2O_5$ from the acid leachate with mixtures of amine and alcohol in a solvent extraction step. Ribas and Nickerson proposed to recover the phosphate values by consolidating, calcining at 870 – 1093°C, and then digesting the clay in mineral acid to produce phosphoric acid. The process was energy intensive and created severe dusting problems during calcination because of the dry, finely-divided particles.

The US Bureau of Mines carried out a detailed investigation in 1982 in which the clay was first sized using hydro-cyclones and hydro-separators. Up to 96% of the plus 400 mesh material was recovered during the sizing step. Subsequent flotation of the plus 400 mesh fraction gave a concentrate of over 30% P_2O_5 at an 88% recovery from this coarse fraction. However, P_2O_5 recovery from the total clay was low.

In 1983, the Florida Institute of Phosphate Research (FIPR) sponsored a project that used an autoclave acidulation technique to recover the phosphate values from clays. At a temperature of 175°C, more than 90% of the P_2O_5 was extracted in less than an hour. After acid treatment, the clay residue quickly consolidated to a 35 – 45% solids level. Longer leaching time led to larger phosphor gypsum crystals, which helped the entire mass to filter more readily. However, the equipment investment and poor acid quality prevented this process from being commercialised.

Other techniques investigated included flocculation, microbial methods and flotation. A common conclusion from most of the previous studies was that recovering phosphate from the slimes is either technically impossible or economically prohibitive.

Pilot plant testing of clays (slimes)

The pilot plant

The pilot work consisted of the chemical conversion of phosphate contained in clays into DCP. The pilot work was performed as a combined effort with PegasusTSI and EcoPhos. The phosphate clays were tested with the EcoPhos technology in a preliminary evaluation phase. The objective was to demonstrate the feasibility of the use of phosphate clays to produce DCP.

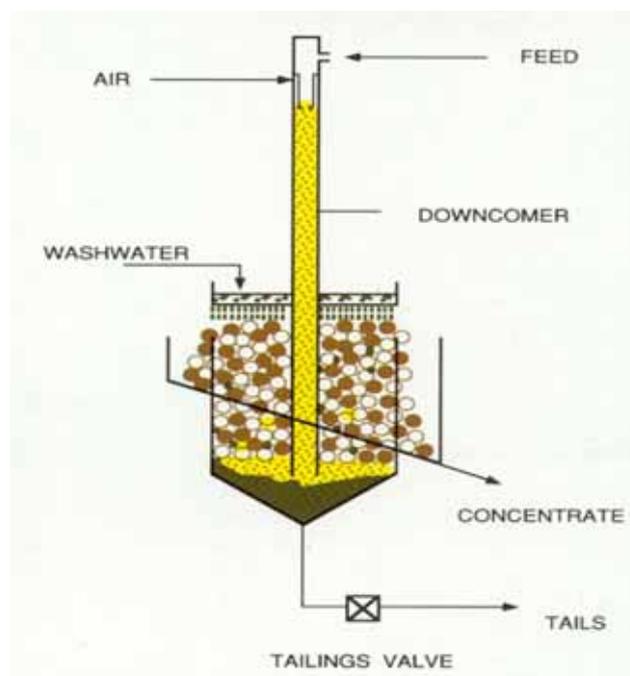


Figure 3. Typical flotation cell.

The process

The core process is based on the phosphate clays attack by diluted hydrochloric acid, followed by a precipitation of the dissolved P_2O_5 as DCP. In this way, the P_2O_5 content of the processed clay is recovered as a solid cake of DCP and the P_2O_5 is concentrated to 42% by precipitation. The chloride coming from HCl is converted into calcium chloride.

X-ray analysis

X-ray analysis was carried out to evaluate the P_2O_5 compounds and assure the solubility of P_2O_5 in HCl. The x-ray analysis showed the presence of the following compounds:

- Brushite: $CaHPO_4 \cdot 2H_2O$.
- Calcium hydrogen phosphate hydrate: $Ca_8H_2(PO_4)_6 \cdot 5H_2O$.
- Gismondine: $CaAl_2Si_2O_8 \cdot 4H_2O$.
- Calcium silicate: $Ca_2(SiO_4)$.
- Calcium hydrogen phosphate iron aluminium silicate hydroxide.
- Kirschsteinite: $Ca(Fe_{0.77}Mg_{0.22})(SiO_4)$.

Results

Available P_2O_5

X-ray analysis showed that most of the phosphate content in the clay was in the form of brushite or calcium hydrogen phosphate hydrate, which are species that are very soluble in HCl. Analysis also showed a high aluminium and iron content, indicating the probable presence of amorphous aluminium and iron phosphate, which are poorly soluble in HCl and could induce some losses linked to insoluble P_2O_5 .

Experimental method

- Step 1: The clays were dissolved in diluted HCl with different HCl concentration at several HCl/Ca ratios. The products were analysed to determine the process yield. The results show that a P_2O_5 recovery of 77.5% can be

➤ Consider the following process:

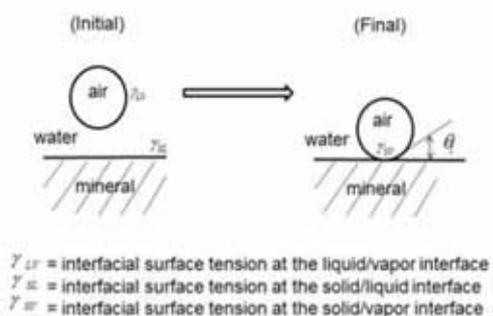


Figure 4. Air mineral contact diagram.



Figure 5. Denver mechanical flotation cell.

Table 1. DCP from slimes

	Unit	Slimes	DCP
P ₂ O ₅	%	13.1	38.3
CaO	%	17.92	36.54
Al ₂ O ₃	%	11.7	0.07
Fe ₂ O ₃	%	8.14	0.18
MgO	%	0.9	0.003
Na ₂ O ₃	%	0.066	0.004
K ₂ O	%	2.65	0.005
As	ppm	7.1	<0.5
Cd	ppm	1.4	<0.5
Cr	ppm	140	1.21
Pb	ppm	3.9	5.5
Sr	ppm	1000	32.91
Hg	ppm	<0.1	0.2
F	%	0.69	0.18

reached based on total P₂O₅ (>90% based on soluble P₂O₅). The results confirm the presence of insoluble P₂O₅ in the clays. Due to the low P₂O₅ in the clays, the quantity of residue was very high. After removal of fluoride, the quantity of solid residue was 0.95 t/t clay input.

- Step 2: The phosphate solution obtained in step 1 was converted into DCP by addition of calcium carbonate. Results showed that for 1 t of clay used, 0.27 t DCP was produced and 0.77 t CaCl₂ effluent solution was generated. The overall quality of DCP was very good.

Conclusion

The batch tests performed shows a P₂O₅ recovery of 76% can be obtained. The DCP obtained is animal feed quality (Table 1).

Flotation process and tailings generation

Flotation overview

Before froth flotation can work, the ore to be treated may need to be reduced to fine particles by crushing and grinding so that the various minerals exist as physically separate grains. This process is known as liberation.

Froth flotation is a highly versatile method for physically separating particles based on the ability of air bubbles to selectively adhere to specific mineral surfaces. The particles with attached air bubbles are then carried to the surface and removed, while the particles that are completely wetted stay in the liquid phase. Chemicals known as collectors can be used to alter mineral surfaces so that they have the necessary properties for separation.

To be effective on a given ore slurry, the collectors are chosen based upon their selective wetting of the types of particles to be separated. A good collector will adsorb, physically or chemically, with one of the types of particles. This provides the thermodynamic requirement for the particles to bind to the surface of a bubble. The wetting activity of a surfactant on a particle can be quantified by measuring the contact angles that the liquid/bubble interface makes with it. Another important measure for attachment of bubbles to particles is induction time. The induction time is the time required for the particle and bubble to rupture the thin film separating the particle and bubble. This rupturing is achieved by the surface forces between the particle and bubble.

The mechanisms for the bubble-particle attachment are very complex, consisting of three steps: collision, attachment and detachment. The collision is achieved by particles being within the collision tube of a bubble and this is affected by the velocity and radius of the bubble. The collision tube corresponds to the region in which a particle will collide with the bubble, with the perimeter of the collision tube corresponding to the grazing trajectory.

The attachment of the particle to the bubble is controlled by the induction time of the particle and bubble. The particle and bubble need to bind and this occurs if the time in which the particle and bubble are in contact with each other is larger than the required induction time. This induction time is affected by the fluid viscosity, particle and bubble size, and the forces between the particle and bubbles.

Table 2. Flotation of tailings – C series reagents

Sample ID Reagent-dosage	Analysis Wt%						%P ₂ O ₅ recovery
	P ₂ O ₅	Insol	MgO	Al ₂ O ₃	Fe ₂ O ₃	CaO	
C1 conc.	31.33	11.96	0.52	0.96	1.72	48.50	32.71
C1 tails	6.60	80.37	0.11	0.27	0.33	10.09	
C2 conc.	29.52	9.74	0.50	0.88	1.52	46.14	82.41
C2 tails	1.92	94.18	0.04	0.12	0.09	3.00	
C3 conc.	30.23	10.17	0.49	0.88	1.64	47.72	76.95
C3 tails	2.63	92.62	0.05	0.14	0.13	3.79	
C4 conc.	29.36	9.93	0.50	0.89	1.57	44.61	69.58
C4 tails	3.25	89.51	0.06	0.17	0.16	5.00	
C4 conc.	28.81	11.45	0.48	0.86	1.52	43.16	80.23
C5 tails	2.22	92.70	0.05	0.14	0.10	3.39	

The detachment of a particle and bubble occurs when the force exerted by the surface tension is exceeded by shear and gravitational forces. These forces are complex and vary within the cell. High shear will be experienced close to the impeller of a mechanical flotation cell and mostly gravitational force in the collection and cleaning zone of a flotation column.

A common quantity used to describe the collection efficiency of a froth flotation process is flotation recovery (R). This quantity incorporates the probabilities of collision and attachment of particles to gas flotation bubbles. The following equation demonstrates this:

$$R = \frac{N_c}{\left(\frac{\pi}{4}\right)(d_p + d_b)^2 H c}$$

Where:

- $N_c = PN_c^i$ is the product of the probability of the particle being collected (P) and the number of possible particle collisions (N_c^i).
- d_p is particle diameter.
- d_b is bubble diameter.
- H is a specified height within the flotation, which the recovery was calculated.



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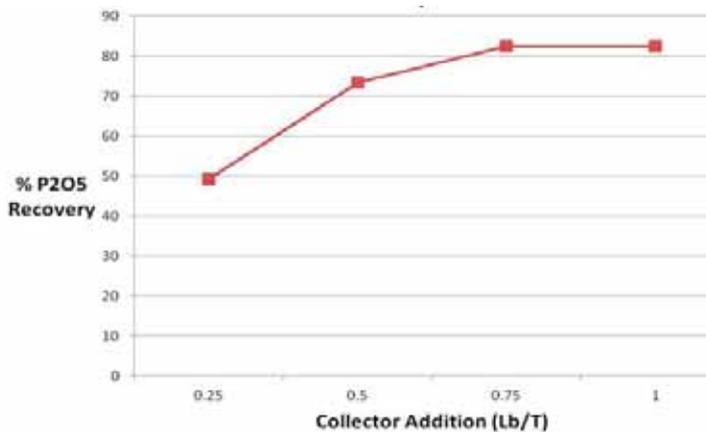


Figure 6. %P₂O₅ recovery versus collector addition.

Table 3. Head sample analysis

P ₂ O ₅	Insol	MgO	Fe ₂ O ₃	Al ₂ O ₃	CaO
2.49	86.11	0.03	0.29	0.28	4.25

Table 4. Pilot plant flotation results

Product	P ₂ O ₅ (%)	P ₂ O ₅ recovery (%)
Concentrate	26.37	82.07
Tailings	1.95	17.93
Weighted average	8.12	100

Figure 4 shows the attachment of the air bubble to the particle.

Tailings generation

The beneficiation process recovers on average 90% of the total P₂O₅ fed to the plant. Typically 30 – 33% of the matrix feed is lost as tailings with a P₂O₅ content of 2 – 6%. The ability to recover the P₂O₅ from the tailings will significantly increase revenue and mine life.

Pilot testing on recovery of mine tailings

The pilot plant testing was a combined effort between PegasusTSI and FIPR. Dr Haijun Liang performed flotation pilot testing with assistance from Shannon Griffin.

Pilot plant

The pilot plant consisted of a Denver mechanical flotation cell with aeration. The collector was a fatty acid (as shown in Figure 5).

Procedure

Various collectors were evaluated to determine which collector had the best recovery. The dosage amount was also adjusted to determine the optimum dosage. Collector C2 with a dosage rate of between 0.75 and 1 lb/t was found to be the optimum collector with an average recovery of 82.41%. The collector was a fatty acid. The results of the collector testing are shown in Table 2.

Once the optimum collector had been chosen, the dosage rate was adjusted to determine the optimum dosage rate for maximum recovery. The optimum dosage rate was found to be between 0.75 and 1 lb/t. The dosage rate versus recovery is shown in Figure 6.

Tailings flotation pilot test results

Once the optimum type of collector and dosage rate was determined, pilot test runs were performed to determine the ability to recover P₂O₅ from the flotation tailings (Tables 3 and 4).

Conclusion

Clays

The batch tests performed shows a P₂O₅ recovery of 76% can be obtained. The DCP obtained is animal feed quality.

Tailings

The pilot plant test results showed that 82.07% of the P₂O₅ in the tailings could be recovered as concentrate for further processing in the chemical plants. The sample initially had 2.49% P₂O₅, and after the flotation, the P₂O₅ in the concentrate was 26.37%.

Economics

Typically a beneficiation plant has a P₂O₅ recovery of about 90%, leaving 10% of the total P₂O₅ lost as clays and tailings. The 10% P₂O₅ loss is typically 7% with the clays and 3% with the tailings. The clay pilot testing showed that 76% P₂O₅ recovery was achieved and tailings pilot work showed that 82.06% P₂O₅ recovery was achieved.

Assuming, 1 million short tpy P₂O₅ production with a 90% P₂O₅ recovery or 10% loss, the 10% P₂O₅ loss was 7% as clays and 3% as tailings. The pilot plant work showed a 76% P₂O₅ recovery from clays and 82.04% P₂O₅ from tailings.

- 1 million short tpy P₂O₅ with 10% loss gives 100 000 tpy P₂O₅ loss.
- 7% as clays 70 000 short tpy P₂O₅ with 76% recovery.
- 53 200 short tpy recovered P₂O₅ converted to DCP.
- 3% as tailings 30 000 short tpy P₂O₅ with 82.04% recovery.
- 24 612 short tpy recovered P₂O₅ converted to DAP.

Profits

- Profit margin from making DCP: US\$12.6 million/yr.*
- Profit margin from making DAP: US\$5.3 million/yr.**
- Total potential profit margin of US\$17.9 million/yr. **WF**

Note

*Assumes US\$100/short t profit on DCP.

**Assumes US\$350/short t DAP sale price and production cost of US\$250/short t, US\$100/short t profit.

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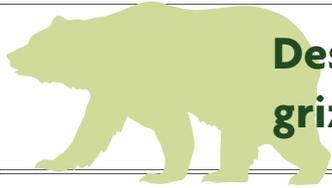


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North America

15 FACTS



Despite being California's official state animal, no grizzly bears have been seen in the state since 1922.

The Mississippi-Missouri River System is the continent's longest river.

Canada owns nearly half of all known global potash reserves.

North America covers **16.5%** of the earth's land area.

Mexico is home to North America's largest wildcat: the jaguar.



The geological makeup of the continent can be dated back to the Proterozoic era.

There are **23** countries in North America.



50% of US urea consumption is in solid fertilizer form.

Although North America is largely self-sufficient with regards to phosphate rock, recently the continent has supplemented resources with imports from Morocco and Peru.

China, Morocco and the US are responsible for **two thirds** of global phosphate production.

The majority of North American phosphate-containing materials are made from wet process phosphoric acid.

After **Asia**, North America is the **second largest producer of sulfuric acid**.

One in every eight Canadian jobs pertains to agriculture.



95% of Canada's potash is exported.

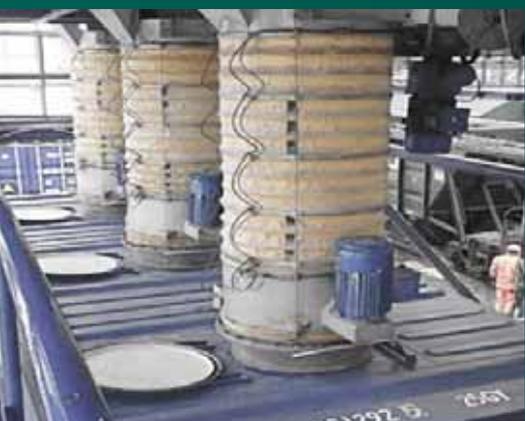
Three oceans border North America: the Atlantic, the Arctic, and the Pacific.

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