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SULPHUR

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New IMO sulphur regulations

Sulphur concrete update

Oxygen enrichment in new SRUs

Sulphuric acid catalysts

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Sulphur concrete

New developments in an old technology.

Oxygen enrichment

Are operators missing out on its benefits?

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Published by: BCInsight

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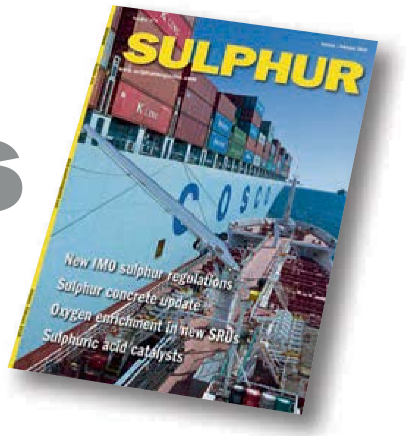
REGULARS

Editorial

China remains the key



> **Equally large if not greater changes are now happening on the demand side of the equation.**

The recent spike in sulphur prices in the third quarter of 2017 seems to have had its origin in Chinese buying. This was in turn driven by lower than expected sulphur supply from Chinese refineries and higher than expected demand from phosphate producers. As can be seen in the market graphs on page 6, sulphur inventories in Chinese ports fell by 800,000 tonnes year on year to August 2017 due to these factors, and low availability was compounded by lower buying on the international market than for 2016. The consequence was that by September prices reacted accordingly as importers – and speculators – raced to catch up.

While prices are now dropping back to more 'natural' levels, it is a salutary reminder that – as eye-catching as developments around the world may be, from Morocco's continual stepping up of phosphate production at the massive Jorf Lasfar complex to the new sour gas-based sulphur coming on-stream in Abu Dhabi, Saudi Arabia, Qatar and Kazakhstan may all be, it is China which continues to hold the key to sulphur markets, and for very good reasons. China remains the leading consumer of sulphur in the world, representing more than 27% of overall demand, and it is also the largest importer, with annual sulphur imports averaging 10-12 million t/a over the past few years, or just under one third of the international market. The most noteworthy developments in previous years have mainly concerned supply side factors, such as the rapid expansion in Chinese sour gas production in Sichuan province, and the wave of new refinery capacity and tightening sulphur regulations in fuels which will see far more sulphur coming from oil in China. Overall, Chinese sulphur production may reach 9 million t/a by 2021, from its present 6.0 million t/a.

However, equally large if not greater changes are now happening on the demand side of the equation. China is undergoing a massive change across the board, as it attempts to manage the transition from a primarily industrial economy to a primarily service-driven economy. It has a rapidly ageing population as huge demographic changes – the result of the 'One Child' policy – finally make themselves felt; far fewer new workers are entering the workforce. This is the primary reason for China's economy slowing from its years of 10%+ growth to the present 6.5%, and this figure may steadily continue to decline over the coming years.

At the same time, public pressure is also forcing the government to tackle issues like pollution and the environment. One major sector where this change is being felt is the fertilizer industry, where the government is trying to force ammonia and urea producers to move away from the coal-based production which has dominated the rise of China's nitrogen industry towards less polluting gas-based plants. Phosphates producers have over-built capacity, and now face both a cap on fertilizer use within China to attempt to deal with over-application and leaching into water courses, and increasingly tight restrictions on airborne emissions which have seen many producers near major population centres forced to close down over winter. Phosphate and other producers are being forced to move at least 10 km away from the Yangtze River as part of a plant by Hubei province to rescue the environment there, affecting Hubei Sanning, Hubei Yihua and Hubei Yangfeng. Taxes on polluting industries may also force less efficient producers to close. The net result is likely to be a continuing decline in China's requirements for sulphur for phosphate production, exacerbated by increased sulphuric acid availability from copper smelters, and a pyrite-based acid industry that has so far resisted the catastrophic decline many had predicted.

All of this makes the decision last year by three of China's largest phosphate producers, YTH, Kailin and Wengfu, to form the TGO sulphur import consortium all the more interesting. If China's sulphur imports start to decline as predicted, these companies, which at present consume 4.5 million t/a of sulphur and which imported 3.1 million t/a in 2016, will come to represent an ever-larger share of the largest import market for sulphur.

Richard Hands, Editor

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Price trends

MARKET INSIGHT

Oliver Hatfield, Director, Fertilizer Research Team, Integer Research (in partnership with ICIS) assesses price trends and the market outlook for sulphur.

SULPHUR

The volatility seen in the sulphur market in October 2017 continued in the last two months of the year. The rapid climb in prices through October and November began to unwind in December. Between the beginning of September and the end of November, the spot price of formed sulphur at most references increased by around $80/t. For several weeks in October, the delivered price to India exceeded $220/t and similar price levels registered on spot business to China. Sulphur prices had not got near that level since February 2014, when they exceeded $210/t for a few weeks, and we must go back to 2011 to find a period when a $210+/t level was sustained for more than a month.

The rate of price inflation and nature of some of the deals agreed as Q4 2017 unfolded suggested a degree of unsustainability, leading us to conclude in our last report that gravity would take hold and bring prices downwards. This turned out to the be the case and, in December, spot sulphur business values lost $30-50/t of their November values. Early in January the delivered to China spot reference was around $155/t, having lost around $50/t in a few weeks, though there were signs that prices would level off at around that level, for at least a week or two.

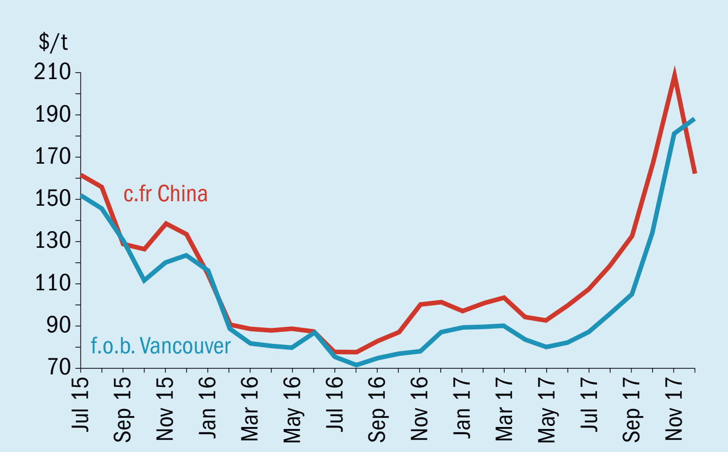
Chinese sulphur business continued to be an important price influence. Stocks of sulphur at Chinese ports which are seen as a leading price indicator had dropped more or less consistently from around 1.8 million tonnes in August 2016 to reach just over 1 million tonnes in August 2017. Stocks have rarely dropped below 1 million tonnes over the last few years, and the decline could be interpreted as Chinese buyers anticipating abundant supply and weaker prices in the last quarter of 2017. The subsequent price rally demonstrated the reverse, and as Chinese spot buyers scrambled to secure product while prices were quickly rising, the doors opened for greater speculative trader activity which probably exaggerated the spike. While year to date Chinese sulphur imports were down 12% in the period to August 2017 compared to 2016, monthly import volumes for September, October and November 2017 were all above year ago levels.

The latest price rally marks another false dawn of the long anticipated sulphur supply tsunami with accompanying sulphur prices on the floor. Ongoing delays at key projects like Kashagan in Kazakhstan which had been expected to lengthen market supply did not materialise in the second half of 2017. Supply availability from the Middle East was relatively tight and this was exacerbated by the start up of Ma'aden's Wa'ad Al-Shamal project. At capacity, this world scale phosphate project requires around 1.5 million t/a of sulphur, and when production at the project was switched on in July 2017 it meant there were fewer export sulphur tonnes available. Monthly prices from the three leading Arab Gulf suppliers spiked in October and November with Saudi Aramco posting November prices at $182/t in November, up $62/t on October, while December prices reached $192/t. Monthly price announcements from Adnoc in the UAE and Qatar Petroleum in Qatar escalated by similar increments.

Not all sulphur markets experienced the same inflation. Price rises for some contract markets in Europe and the US, which are more insulated from developments in spot formed sulphur, were of a significantly smaller order of magnitude. In Europe, the quarterly contract price of liquid sulphur delivered to northwest Europe changed relatively little and finished 2017 at the equivalent of around $100/t, a similar level to the start of the year. At Tampa, the Q4 2017 contract price increased but by just $36/ton, to reach just shy of $110/ton.

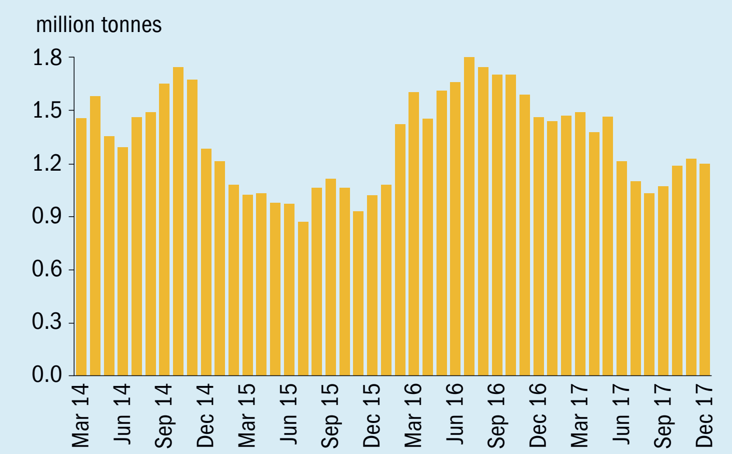
Not surprisingly, given the speed and scale of the increase in sulphur prices, many sulphur buyers found it difficult to absorb higher sulphur costs and secure sulphur volumes. OCP of Morocco, which buys more than 5 million tonnes of sulphur each year, was notably short of product in the last quarter of 2017. Sulphur export availability from Russia, one of OCP's key suppliers, was short after Austrofin Gazprom was forced to cancel Q4 contracts due to weather related disruptions to Russian waterways. Consequently, OCP looked to the spot market to source additional sulphur tonnes, and sought increased imports of sulphuric acid. For a few weeks, there were rumours that OCP might delay by six months the start up of its fourth phase phosphate unit, which will add around 500,000 tonnes of additional

Fig. 1: **Month average spot sulphur prices, Jul 15 to Dec 17**



Source: Integer, ICIS

Fig. 2: **China sulphur port inventory, 2014 to 2017**



Source: Integer, GTIS

sulphur import demand, in response. However, this turned out to be unfounded, and the plant is expected to stick to the schedule of first production in Q1 2017. In South Africa, one of the highest cost phosphate producers, Foskor, announced in November that it would idle phosphate operations until further notice, in response to high sulphur prices and weak phosphate margins.

SULPHURIC ACID

Sulphuric acid prices were generally higher in the last two months of 2017 as the balance between supply and demand in most markets was either closely matched or tight. Most of the countries that had released data for the year to date period to November 2017, recorded higher acid imports compared to the same period in 2016, while many significant exporting countries saw volumes contract. Exports from two of the leading countries, Japan and Korea, have been depressed by planned outages at metallurgical acid plants. Meanwhile, the dramatic fly-up in sulphur prices also supported sulphuric acid prices, indirectly.

In Europe acid market price sentiment favoured sellers over buyers. In the second half of December, f.o.b Mediterranean acid prices increased from the low $20s/t to around $30/t, with the f.o.b NW European price following the same trajectory. With robust sulphur prices, there was no room for producers of burner acid to alleviate any acid supply shortages. Expectations for 2018 contracts are that prices would move upwards, with an increase of around €5/t over 2017 expected. The first half of the year is seen as being particularly tight in part due to lost production. On the supply side, Aurubis announced that it will undergo a two year inspection shutdown which will last throughout the second quarter of 2018.

The impact of a tight sulphur market was keenly felt in the acid market in Africa. OCP, Africa's biggest maker and buyer of sulphuric acid, faced a predicament after a key Russian sulphur supplier declared force majeure on Q4 volumes as harsh weather conditions prevented sulphur getting to export port. OCP turned not only to other sulphur sources but also to the sulphuric acid market. Having already seen import volumes for the year to date period to October 2017 reach 1.2 million tonnes, about 5% ahead of the prior year period, OCP was reported to have booked shipments of approaching 190,000 tonnes of sulphuric acid for December 2017, compared to 108,000 tonnes in 2016.

Copper prices supported operating rates for copper producers. However, labour disputes led to a reduction in sulphuric acid supply temporarily in Chile, with Southern Copper and Enami reporting short term strikes in December, which were reported to be resolved by January. Some spot cargoes of sulphuric acid were reported delivered to Chile at values of $80/t and possibly even much higher (see China below) in December. This no doubt coloured discussions about 2018 annual contract values. In October consensus was that prices in the high $60s might be agreed, but by November and early December discussions had moved to a higher range, with buyers offering $65-75/t versus seller ideas above $80/t, with prices being talked up in part due to higher freight rates. Some deals were reported done as high as $85/t, but most agreements were reported in the $70s.

Elsewhere, production and export volumes were generally below normal levels in some important locations. In the US, Rio Tinto lifted a force majeure in early January 2018 at its Kennecott operation which had been imposed in October 2017. With less acid available there were reports that volumes would be singularly dedicated to supplying Agrium which normally takes around two thirds of output. Ongoing maintenance at key sulphuric acid producers in Japan (Sumitomo, Mitsubishi, and Pan Pacific Copper) continued in November, though some operations returned to normal in December. On the other hand, despite the dramatic rise in the cost of imported sulphur to China, there were reports that Chinese virgin acid seller Two Lions found a market for three cargoes in December and January, thought to be for the Chilean market. No price information was reported, but with a delivered China sulphur price of $150/t or higher, this business would likely need to achieve a delivered Chile sulphuric acid price of around $100/t to make sense, assuming freight at market rates. ■

Price indications

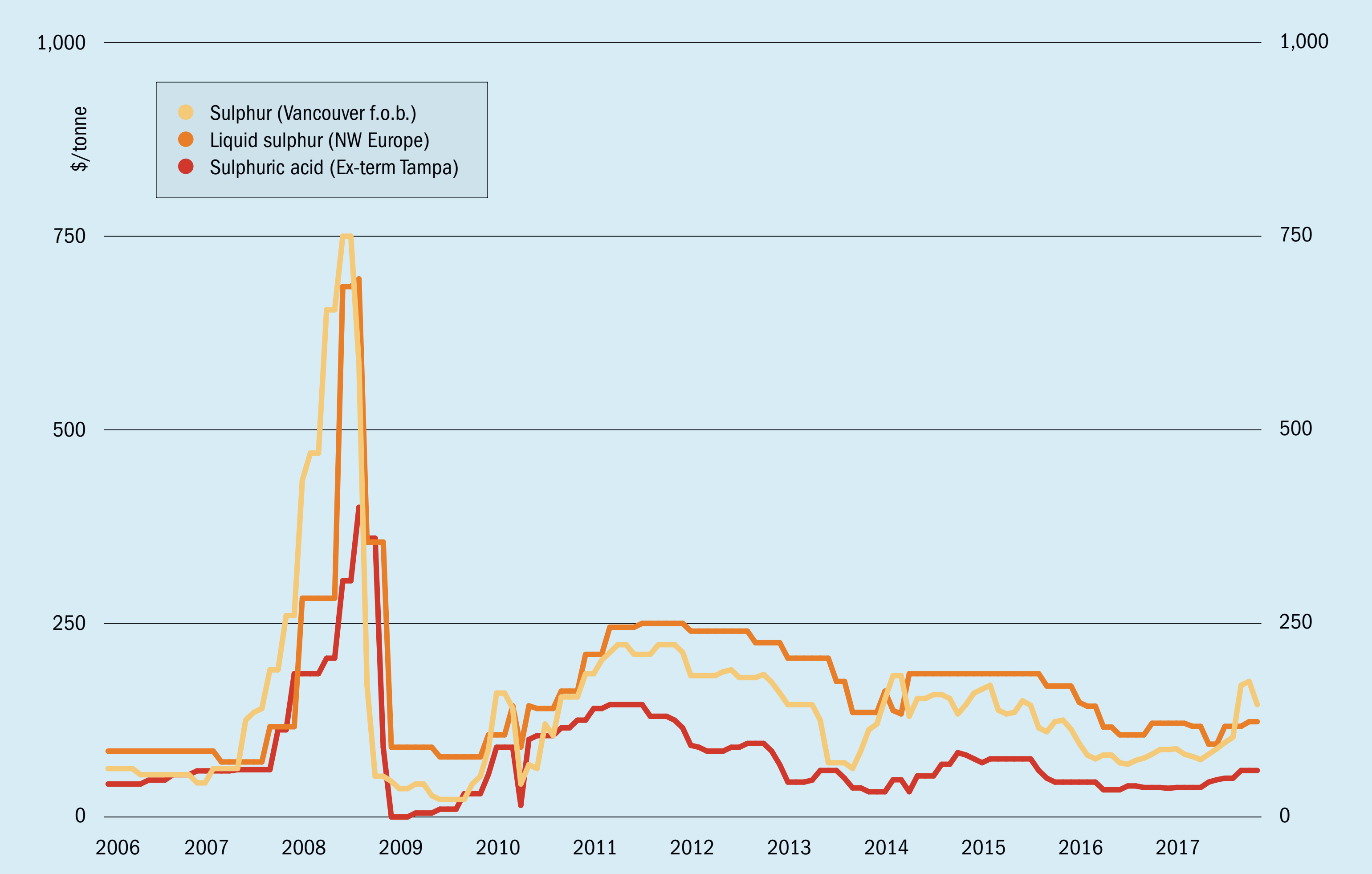
Table 1: **Recent sulphur prices, major markets**

Cash equivalent	August	September	October	November	December
Sulphur, bulk ($/t)					
Vancouver f.o.b. spot	96	103	170	175	145
Adnoc monthly contract	102	110	127	184	195
China c.fr. spot	116	135	185	190	150
Liquid sulphur ($/t)					
Tampa f.o.b. contract	74	74	74	110	110
NW Europe c.fr.	117	117	117	123	123
Sulphuric acid ($/t)					
US Gulf spot	50	50	60	60	60

Source: various

Market outlook

Historical price trends $/tonne



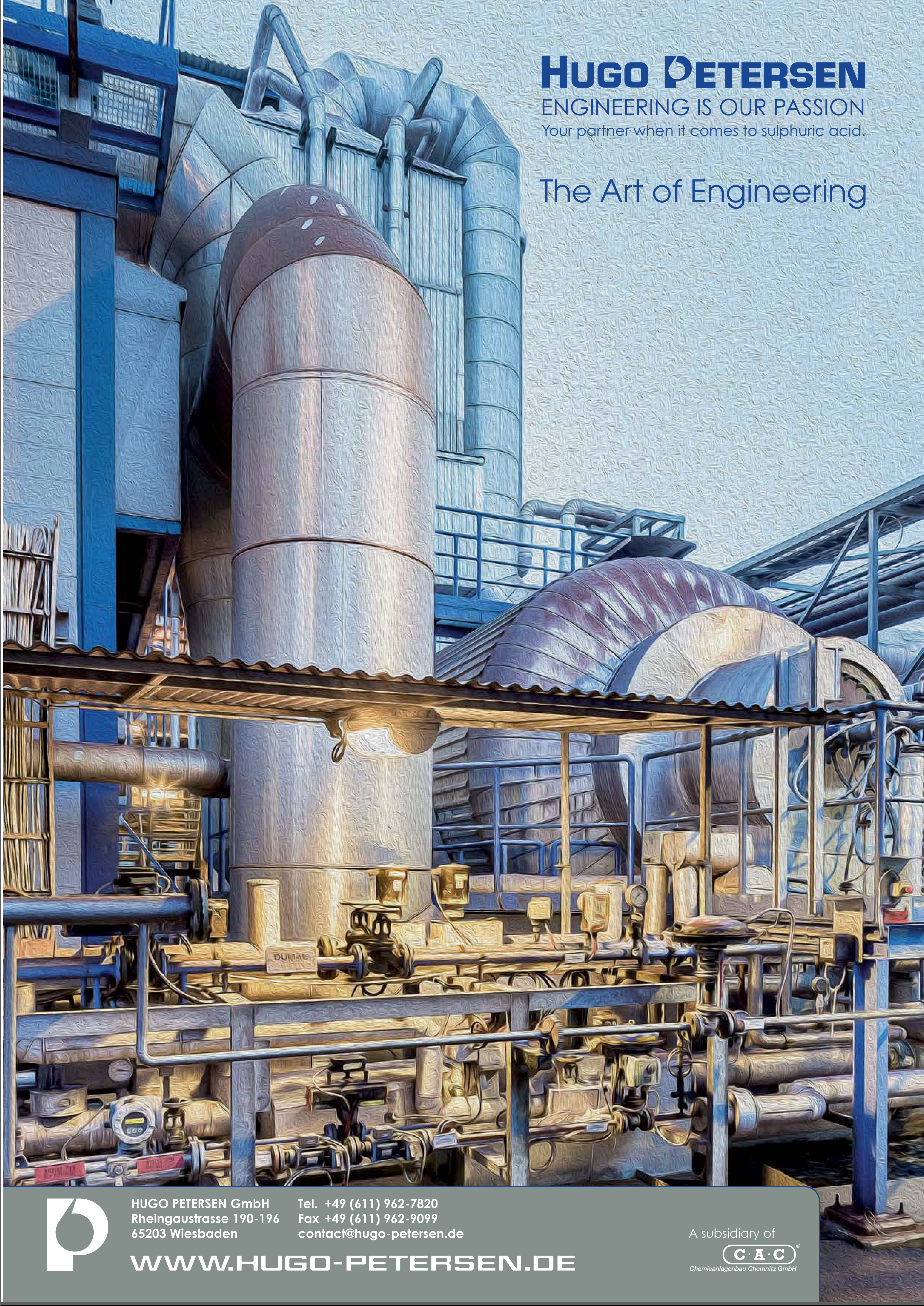
Source: BCInsight

SULPHUR

- In our last outlook our view was that in spite of conflicting market signals, prices would likely soften toward the end of 2017, and that materialised with significant price corrections registered in December. However, predicting price direction for the first few months of 2018 is a greater challenge than usual as the picture remains cloudy.
- In China, although port stocks built up due to relatively robust import volumes in October through early December 2017, there was a reversal thereafter and in the first week of January 2018 stocks were reported to have fallen to 1 million tonnes once again. Underlying demand for sulphur from the Chinese phosphate sector is generally seasonally high through the first quarter of the year, suggesting that the Chinese market will remain tight.
- Export availability from Russia should improve seasonally as we approach the spring, correcting one of the contributing factors to the Q4 2017 price fly-up, but this is unlikely to correct until the latter part of Q1.
- OCP is likely to be looking for additional sulphur tonnes as it starts up its latest phosphate processing unit in March 2018.
- Looking beyond 1Q 2018, the fundamentals point to sulphur supply growing substantially faster than demand and other things being equal, we would expect the sulphur market to weaken significantly. New projects include expansion at the Reliance operation in India which is set to add 600,000 t/a of sulphur at capacity. On a larger scale is the Kashagan project in Kazakhstan which has the potential to add 1.2 million t/a, but this project has consistently missed its production targets.

SULPHURIC ACID

- It looks likely that the finely balanced to tight market conditions will persist through January 2018. Thereafter, we would expect to see restoration of relatively normal supply volumes. Since reduced supplier availability has been an important contributor to the recent increase in acid prices, we would therefore expect to see prices soften.
- For the year to date to November 2017, Japanese exports of sulphuric acid totalled 2.4 million tonnes, compared to 2.8 million tonnes the previous year, due to maintenance lasting between 25-40 days at three of the country's largest sulphuric acid producers in October to December, with combined sulphuric acid capacity of around 6.5 million t/a. So we would expect Japanese export availability to increase in January 2018 and beyond. Tonnage was also lost in the last quarter of 2017 in the Philippines, US and South America due to technical interruptions and labour unrest, and supply should be up in these locations in 2018, assuming these issues are resolved and not repeated.
- It is also possible that the support for the acid market which has come from the fly-up in sulphur prices in the last quarter of 2017 will dissipate, but this is dependent on the timing of projects scheduled to add new sulphur supply.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QATAR

Muntajat to market Qatari sulphur

The Qatar Chemical and Petrochemical Marketing and Distribution Company (Muntajat) is expanding its marketing, sales and distribution activities by adding sulphur to its product portfolio. Established in 2012, Muntajat is a state-owned company which serves as the exclusive distributor of over 13.6 million t/a of chemical and petrochemical products from Qatar, and now numbers 3,000 customers in 135 different countries. The inclusion of Qatar's sulphur output will occur as from January 1st 2018. Qatar currently produces and exports more than 2.3 million t/a of sulphur from its refineries and gas processing sites, mainly the Common Sulphur Facility at the Ras Laffan LNG/GTL site. This is expected to rise to 4 million t/a by 2020 when Qatar reaches its production target of 100 million t/a of LNG.

The company said in a statement that; "bringing sulphur into our fertiliser product portfolio supports the increased demand we are seeing from fast growing market segments, particularly the fertiliser industry. It also underscores our ongoing commitment to securing supply for our global customers, giving them access to a wider range of products from one supplier. With the expansion of sulphur to our portfolio, we are better positioned than ever to respond to rapidly growing demand, enabling customers to deliver the materials and solutions they need for the markets they serve." ■

KUWAIT

First phase of sulphur forming project completed

The Kuwait National Petroleum Company (KNPC) says that the construction of the first phase of its liquid sulphur treatment project at the Mina al-Ahmadi refinery has been completed. The facility includes four storage tanks for liquid sulphur, with a total capacity of 18,000 tonnes, as well as 5,000 t/d of granulation facilities and a warehouse with a capacity of 145,000 tonnes of solid sulphur, together with a jetty for loading and export of the sulphur. It is Kuwait's first facility for the production, storage and export of solid sulphur, and will handle liquid sulphur from the KNPC Clean Fuels Project and facilities of the Kuwait Oil Co. The Mina al-Ahmadi refinery processes 440,000 bbl/d of oil. The first sulphur shipment left the new facility in early November 2017 according to KNPC.

Phase 2 will expand liquid sulphur storage to 340,000 tonnes and solid sulphur forming capacity will increase by 3,000 t/d to 8,000 t/d. Solid sulphur storage capacity will expand to 235,000 tonnes. Kuwait's sulphur export/loading capacity will reach 60,000 t/d. The total cost of the facility is put at $96 million.

GERMANY

Clariant to supply RTI's warm gas desulphurisation technology

Clariant has announced the signing of a global licensing agreement with RTI International granting Clariant exclusive right to supply their solid sorbent material, vital for RTI's warm gas desulphurisation (WDP) process technology. WDP enables sulphur-containing gas streams, such as synthesis gas from coal or petroleum coke gasification, to be cleaned at elevated temperatures (250-650°C), thus reducing or eliminating the need for substantial gas cooling and expensive heat recovery. This increases overall process efficiency, reduces greenhouse gas emissions, and reduces the capital and operating costs of the entire gas clean-up block by up to 50% compared to conventional technologies.

WDP technology uses a novel transport reactor design and a unique high capacity, regenerable solid fluidisable sorbent (supplied by Clariant). Able to function across a wide range of operating temperatures and pressures, the sorbent has a high capacity for adsorbing sulphur, removing H_2S and COS to very low levels and allowing customers to treat large gas stream volumes. It is regenerable with a low attrition rate and capable of long cycle lengths without major replacement requirements, reducing the need for shutdowns. The technology can achieve up to 99.9% removal of total sulphur from syngas at temperatures as high as 650°C and over a wide range of sulphur concentrations. Integration of this technology with a downstream activated-amine carbon capture process enables further reduction of total sulphur in the syngas to sub-ppmv concentrations (as low as 100 ppb), suitable for stringent synthesis gas applications such as chemicals, fertilizers, and fuels.

CHINA

Sinopec awards contract for five acid alkylation units

DuPont Clean Technologies has signed contracts with China Petroleum & Chemical Corporation (Sinopec) for five grassroots *STRATCO*® alkylation units at five Sinopec refineries in China. The scope of the contracts includes the license, engineering and supply of proprietary equipment for Sinopec Yangzi Company (YPC) – one of China's leading suppliers of olefins and aromatics – as well as the Sinopec Zhenhai Refining and Chemical Company (ZRCC), Sinopec Tianjin, Sinopec Qilu and Sinopec Zhongke.

Sinopec is looking to comply with strict gasoline emissions regulations introduced as part of the China V standards in January 2017. The alkylation technology enables refiners to produce cleaner-burning fuel with higher octane and extremely low sulphur content, low Rvp and zero olefins. The five units commissioned by Sinopec range in size from 300,000 t/a (7,700 bbl/d) to 400,000 (10,300 bbl/d) of alkylate production. Start-up for the first four alkylation units is expected by mid to late 2018.

"With more than 170 million vehicles on the road, China will adopt even tougher National VI emission standards by July 2020," said Eli Ben-Shoshan, global business director, DuPont Clean Technologies. "We are delighted to be able to help Sinopec ensure its refineries are ready to meet strict fuel requirements with our *STRATCO*® alkylation technology."

UNITED ARAB EMIRATES

Inauguration of Habshan expansion

Sheikh Hamdan bin Zayed Al Nahyan officially inaugurated the most recent expansion to Adnoc's Habshan 5 gas processing plant in November 2017. The plant, linked via a 215 km pipeline to Adnoc's offshore Umm Shaif gas field, has four gas processing trains and sulphur recovery units, with a daily production capacity is 12,000 t/d of natural gas liquids, 1.1 billion scf/d of sales gas; 5,200 t/d of liquid sulphur and 3,000 bbl/d of condensate. The expansion has taken gas processing capacity to 2.3 billion scf/d, or 110% of original capacity, enabling the plant to process an additional 134 million scf/d. As part of Adnoc's Integrated Gas Development Expansion project, Habshan 5's gas processing capacity will increase by 20%, or a further 400 million scf/d, in the second half of 2018. It is the first Adnoc gas processing facility to be designed with integrated flare gas recovery units to reduce its environmental impact. It can extract more than 99% of natural gas liquids, to maximise value, and its sulphur recovery units are integrated with tail gas treatment units to improve sulphur recovery to up to 99.99%.

Adnoc awards offshore sour gas FEED contracts

Adnoc has awarded the two front end engineering design (FEED) contracts for the company's planned massive offshore sour gas project, consisting of the Hail, Ghasha and Dalma fields. Bechtel (UK) was awarded the Hail & Ghasha FEED contract and TechnipFMC the Dalma FEED contract. These are some of the largest FEED contracts in terms of man-hours ever awarded by an oil and gas company, highlighting how critical a detailed engineering study is in optimising the project schedule and cost. In addition to the FEED contracts, Adnoc is reportedly close to awarding five technology licensor contracts, covering gas treatment; a sulphur recovery unit (SRU); natural gas liquids; condensates recovery and hydrogen generation. Hail, Gasha and Dalma are estimated to collectively hold trillions of cubic feet of recoverable gas. The overall project is expected to produce more than 1 billion cfd of sales gas, sufficient to generate electricity to power two million homes in the rapidly growing Emirate.

Sultan Ahmed Al Jaber, UAE Minister of State and ADnoc Group CEO, said: "The growth in energy demand in Abu Dhabi, and the wider UAE, has prompted Adnoc to further harness its gas resources, as part of its 2030 smart growth strategy. This FEED award provides Adnoc with the potential to unlock additional undeveloped sour gas reserves and will allow us to deliver against our strategic objective to ensure a sustainable and economic supply of gas."

Sulphuric acid production at the Jorf Lasfar phosphate hub.

Abdulmunim Saif Al Kindy, director of Adnoc's upstream business, said: "The decision to award both FEED contracts came after a rigorous and extremely competitive tendering process, ensuring we will strictly manage costs by working with contractors that can deploy effective engineering and robust value-add technologies. In progressing with these projects, we create the potential to capitalise on our success and experience in ultra-sour gas production, gained from the development of the Shah field, the largest project of its kind in the world."

MOROCCO

OCP signs sulphur supply deal with Adnoc

OCP, the world's largest phosphate producer, has signed a long-term sulphur supply contract with Adnoc, the world's largest sulphur exporter. The contract will run to 2025, with a steady increment in supply from Abu Dhabi to Morocco over that time. Morocco imported 2 million t/a of sulphur from Adnoc during 2016.

Both companies emphasised the synergies between OCP – currently rapidly expanding its phosphate production – and Adnoc, whose massive expansion in sour gas production to achieve self-sufficiency in gas output is generating huge volumes of sulphur.

Abdulla Salem Al Dhaheri, Marketing, Sales and Trading Director at Adnoc, said: "This landmark agreement, which is unique in the sulphur industry, strengthens Adnoc's position as one of the world's largest exporters of sulphur. It will reinforce the sustainable supply of sulphur to Morocco and enhance our ability to achieve positive margins."

Mustapha El Ouafi, Managing Director at OCP, said: "since 2008, OCP has initiated the largest investment program in the fertilizer industry with the objective of doubling its mining capacity and tripling its fertilizer capacity. Our ambitious program will see OCP further strengthen its position as the world's largest fertilizer producer and a leading player in the agribusiness value chain. As such, we are committed to further developing a reliable and strategic partnership with ADNOC, the world's largest sulphur exporter."

CANADA

Agrium and PotashCorp merge

Agrium and PotashCorp successfully completed their merger at the beginning of January.

Nutrien, the new company created by the merger, will be the world's largest fertilizer manufacturer and retailer. It will be a massive international player with nearly 20,000 employees and operations and investments in some 14 countries.

The proposed Agrium-PotashCorp merger was originally unveiled in September 2016, with the unanimous blessing of the boards of both companies, and promised to create a new fertilizer sector giant valued at around $36 billion. The so-called "merger of equals" was subsequently subject to a drawn-out regulatory review and approval process in Brazil, Canada, China, India, Russia and the US.

After 15 long months, the merger finally received the all-clear and overcame its last hurdle with the regulatory approval of the US government in late December 2017

Confirmation of the merger's success came from Chuck Magro, Nutrien's new president & CEO:

"Today we are proud to launch Nutrien, a company that will forge a unique position within the agriculture industry. Our company will have an unmatched capability to respond to customer and market opportunities, focusing on innovation and growth across our retail and crop nutrient businesses. Importantly, we intend to draw upon the depth of our combined talent and best practices to build a new company that is stronger and better equipped to create value for all our stakeholders."

To gain regulatory approval, Potash Corp has agreed to divest itself of its stakes in rival potash producers SQM, Arab Potash (APC), and Israel Chemicals Limited (ICL). Agrium also divested its US nitric acid and phosphate production assets (*Fertilizer International* 481, p 10).

Despite these sell-offs, Nutrien still emerges as the world's largest standalone fertilizer producer, selling over 25 million tonnes of potash, nitrogen and phosphate products annually – into worldwide agricultural, industrial and feed markets.

Notably, Nutrien will control a massive 22 million t/a of Canadian potash production capacity. This is combined with almost 11 million t/a of tonnes of nitrogen production capacity, making it the third-largest nitrogen fertilizer producer globally. Its phosphates operations, by adding a further 4.3 million t/a of production capacity, also make Nutrien North America's second-largest phosphates producer.

Importantly, the new company's manufacturing might is married to equally impressive retail reach. Nutrien has come into possession, via Agrium, of the world's largest agricultural retail network, spread across some 1,500 locations in North America, Australia, and South America. This network is capable of generating around $12 billion in annual sales. Nutrien also gains global distribution and market access for its potash output through its participation in Canpotex, Canada's highly successful potash export partnership.

Nutrien began trading on the Toronto Stock Exchange and the New York Stock Exchange on 2 January under the ticker symbol NTR.

Chuck Magro highlighted some of Nutrien's immediate priorities in a video message on the company's website: "2018 will be our first full year, and of course we have ambitious plans. We made a public commitment when we announced the deal to deliver $500m of annual operating synergies. There will [also] be a strong focus to grow the retail business in North America, but we also have plans to grow the network in Brazil."

Nutrien has committed itself to cutting its annual operating costs by $500 million by the end of 2019. This includes initial savings of $250 million this year. These will be delivered through distribution and retail integration, procurement savings and optimisation of production and SG&A.

BAHRAIN

BAPCO refinery upgrade deal awarded

The state-owned Bahrain Petroleum Co. (Bapco) has awarded the main contract to expand and upgrade the kingdom's refinery. A consortium of France's TechnipFMC, South Korea's Samsung Engineering and Spain's Tecnicas Reunidas was announced on December 4th as the winner of the $4.2 billion lump sum turnkey engineering, procurement, construction and commissioning (EPCC) contract to expand the 79-year-old refinery at Sitra, in the kingdom's north-east from 267,000 bbl/d of processing capacity to 360,000 bbl/d, as well as adding units for the production of cleaner, lighter, higher-value fuels, predominantly for export. Facilities to be added under the Bapco Modernisation Programme include residue hydrocracking, hydrocracking, hydrodesulphurisation, crude and vacuum distillation, hydrogen production, hydrogen and sulphur recovery, tail gas treatment, sour water stripping, amine recovery, bulk acid gas removal and amine recovery units, as well as addition facilities for recovery, solidification and handling of sulphur. Completion is scheduled for 2022.

With Bahrain's own oil reserves running down, the Bapco refinery is primarily supplied by pipeline from neighbouring Saudi Arabia, and a key component of the expansion will be the associated expansion in pipeline capacity from the Abqaiq processing hub in Saudi Arabia from 230,000 bbl/d to 350,000 bbl/d, due in 2018. While financial challenges remain for the project after Bahrain suffered a credit rating downgrade in November, the participation of Samsung is expected to lead to financing from South Korean loan agencies.

INDIA

India looks to reduce petcoke imports unless sulphur emissions are curbed

India's petroleum minister Dharmendra Pradhan says that the government is working to curb India's imports and use of petroleum coke. The plan is to only allow the use of pecoke in sectors which absorb the sulphur emissions in the manufacturing process, such as the cement industry and gasification plants, to reduce sulphur emissions to atmosphere. India's imports of petcoke have soared from 3.3 million t/a in 2012-13 to 14.4 million t/a in 2016-17, and total national consumption reached 23.25 million t/a that year due to its use in power generation. Reliance Industries has also recently brought on-line a massive $4.6 billion petcoke gasification plant at its Jamnagar refinery complex, which will produce up to 2,000 t/d of sulphur extracted from the gasification process.

UNITED STATES

Axens completes catalyst plant upgrade

Axens has completes the expansion of its Calvert City, Kentucky catalyst plant. The facility will now produce the company's full range of Impulse™ hydroprocessing catalysts in North America. Impulse is a range of high performance hydrotreating catalysts covering proesses from naphtha to vacuum gasoil (VGO) hydrotreating and hydrocracker pretreatment which Axens claims offers higher flexibility and maximum throughput allowing operators to process more difficult feedstocks with higher end boiling points and longer cycles. This is one of several sites with the capability to meet the global demand for these catalysts.

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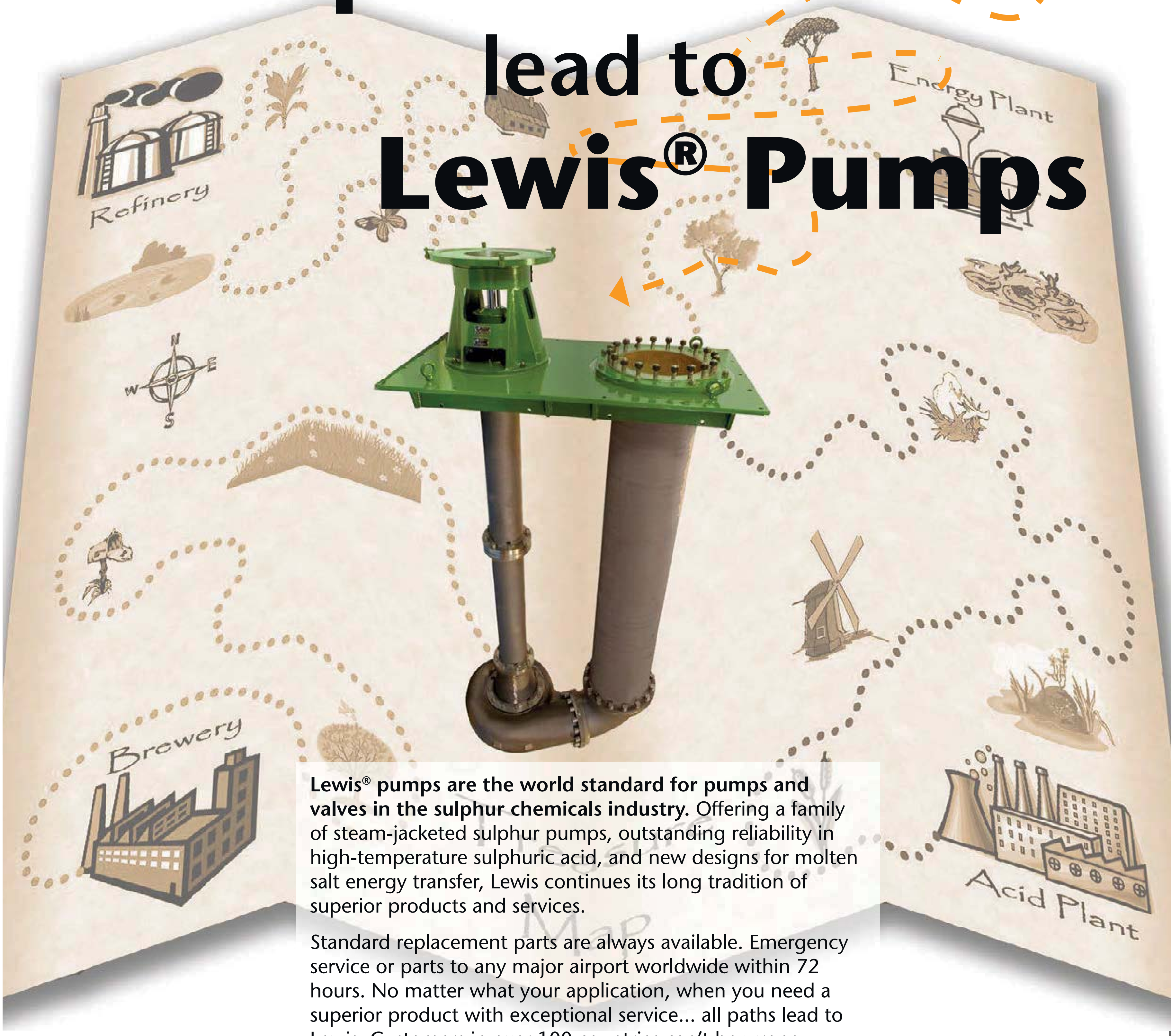

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RUSSIA

Acron to build new sulphur-burning acid-plant

DuPont Clean Technologies says that it has been awarded a contract by PJSC Acron to license and provide engineering services and proprietary equipment for a new *MECS®* sulphur-burning sulphuric acid plant. The plant will have a design capacity of 2,100 t/d and will use the energy efficient MECS Heat Recovery System (*HRS*™). It will be a part of a new phosphate fertilizer complex, which Acron plans to build at the existing Dorogobuzh production site in the Smolensk region of Russia. The detailed design of the fertilizer complex will be carried out by Acron's own subsidiary, LLC Novgorodskiy GIAP. As well as sulphuric acid, the complex will produce phosphoric acid, MAP, DAP and NPK fertilizers. Long-time DuPont partner SNC-Lavalin will provide the design packages and other licenses and services for the new fertilizer project. Construction of the facility and its start-up are due to be completed in Q4 2020, with installation of the HRS equipment and sulphuric acid production set to begin in 2019.

Ivan Antonov of Akron (left) and DuPont Clean Tech's Eli Ben-Shoshan at the signing of the agreement to build Acron's new sulphuric acid plant.

"Our aim in using MECS services and technology in our design is to enable the Dorogobuzh facility not only to comply with environmental requirements on sulphur dioxide emissions, but to also recover maximum energy with minimal corrosion or maintenance using the HRS technology," said Andrei Kolosovsky, CEO of LLC Novgrodoskiy GIAP. "We take our responsibility to the environment and the communities in which we operate seriously. The technology will allow us to be a good corporate citizen while saving energy and running the sulphuric acid plant efficiently."

ZIMBABWE

Zimphos revamp nearing completion

Zimphos says that it is on course for completion a $7.5 million revamp of its phosphate processing activities. The company, a subsidiary of the state Industrial Development Corporation (IDC), has been producing only 100,000 t/a of phosphate fertilizer against a notional capacity of 250,000 t/a, and in 2014 produced only 20,000 tonnes while its phosphoric acid plant was offline for refurbishment. However, with $5 million of investment money already committed and a further $2.2 million to come from the Reserve Bank of Zimbabwe, the majority of revamp work has now been completed. Operations have been assisted by the recapitalisation of fellow IDC subsidiary Dorowa Minerals, which mines and beneficiates phosphate rock in the Upper Save valley 90km west of Mutare.

Zimphos says that the bulk of the $7.5 million ($4.2 million) is being spent on rejuvenating the sulphuric acid plant, and a further $1.5 million on the phosphoric acid plant and $600,000 on the triple superphosphate (TSP) plant. Other money will go on the aluminium sulphate plant, materials handling equipment, and other structures and utilities.

DEMOCRATIC REPUBLIC OF CONGO

New acid plant for Katanga

Katanga Mining Ltd says that it has successfully completed hot commissioning of the core of the first train of its new whole ore leach processing facility at its subsidiary Kamoto Copper Company's copper and cobalt mine in Lualaba Province. The site where the leach and electro-winning plants are located successfully produced its first copper on December 11th. Copper and cobalt production had been suspended since September 2015 pending the construction of the ore leach project. A progressive ramp-up and commissioning of the remainder of the first train is expected to follow over the next three months, with full capacity scheduled for the end of Q1 2018.

Johnny Blizzard, Chief Executive Officer of Katanga, commented: "We are very pleased to have met our anticipated budget and timetable for commissioning the first train of our new plant and are optimistic that the tangible improvements from using a whole ore leach processing circuit will be seen in the near future. We look forward to ramping up to full production capacity of the first train. The construction of the second train is also on schedule and budget and hot commissioning is still expected to commence in H2 2018."

Katanga says that its board has also approved capital budgets for the engineering and construction of an upgraded cobalt processing plant and a sulphuric acid production plant at KCC. The company will spend $15.8 million to engineer and construct a facility designed to reduce throughput bottlenecks in its existing cobalt processing circuit to align with the life of mine cobalt production plan of 30,000 t/a average annual production, and $237 million spread over 2018 and 2019 to construct a sulphuric acid and sulphur dioxide production plant at KCC. This will improve the reliability of the supply of these reagents to the ore processing circuit. The acid plant is designed to produce 1,900 t/d of sulphuric acid, 200 t/d of sulphur dioxide and 17MW of co-generated power, reducing KCC's reliance on imported reagents. Commissioning of this plant is expected to commence in H2 2019.

UNITED STATES

Veolia to debottleneck acid regeneration facility

Veolia says that it plans to expand sulphuric acid regeneration capacity at its Burnside plant at Darrow, Louisiana. The Burnside facility regenerates spent sulphuric acid from local refineries and other customers and has been in continuous operation for 50 years. It is Veolia's largest hybrid sulphur burning facility, capable of handling both fuming and non-fuming acids, and shipping 15-25 truckloads of sulphuric acid to customers every day. The debottlenecking project will increase spent sulphuric acid regeneration capacity by 15% annually and is expected to be completed during the 3Q 2018 turnaround. President and CEO Bill DiCroce said this is an important step forward in growing the plant's regeneration services capabilities. Refiners are pushing the current acid regeneration circuit to nearly 100% capacity because of spiking demand. By adding capacity through this expansion project, Veolia says that is supporting its customers' growth requirements as well as positioning itself for further growth.

Itafos to buy Agrium Conda operations

Itafos says that it has signed a definitive purchase agreement with Agrium Inc. to acquire Agrium's Conda Phosphate Operations, an integrated producer of phosphate fertilizers and specialty products, for $100 million (including inventory) on a cash and debt-free basis. Conda Phosphate Operations, located in Conda, Idaho, includes phosphate production facilities and adjacent phosphate mineral rights. It produces approximately 540,000 t/a of monoammonium phosphate, super phosphoric acid, merchant grade phosphoric acid and specialty products and serves the North American fertilizer market.

The transaction includes long-term strategic supply and off-take agreements, under which Agrium will supply 100% of the ammonia requirements of Conda Phosphate Operations and purchase 100% of MAP product produced, with pricing formulas for both tied to benchmark phosphate fertilizer prices.

"This transaction is transformative for Itafos and vastly accelerates our strategic objective of becoming a leading global player in the phosphate fertilizer industry," said Brian Zatarain, CEO of Itafos. "Conda Phosphate Operations further diversifies our global position of long-term strategic phosphate assets with an operating business in North America that has a long and successful track record of safe, responsible, reliable, continuous and financially stable operations."

The transaction is expected to close by year end 2017, subject to customary closing conditions, including approval of the Federal Trade Commission.

ETHIOPIA

Yara signs SOP mining agreement

Yara has signed a mining agreement with the Ethiopian authorities, making possible the future development of the Yara Dallol potash mine. The signing ceremony took place in November in Addis Ababa, and was attended by Ethiopian Minister of Mines, Petroleum and Natural Gas, Ato Motuma Mekasa, and Yara International president and CEO, Svein Tore Holsether.

Yara Dallol is a mining project located in the Afar region in the northern part of Ethiopia. During the feasibility studies carried out over the recent years, significant reserves of natural resources used for the production of sulphate of potash (SOP) have been identified in the allocated exploration area. SOP is especially beneficial for fruit, vegetable and coffee crops. The planned mine will have a capacity of approximately 600,000 t/a of SOP, equivalent to approximately 10% of the global market. The products will be mined using solution mining, meaning there will be no open pit at the site.

Yara Dallol is a 51.8% owned by Yara International, together with Liberty Metals and Mining Holdings (25%) and XLR Capital (23.2%). A final investment decision is expected towards the end of 2018. The total capital expenditure for the project has yet to be finalised, but is likely to be lower than the previous estimate of $740 million, according to Yara.

DENMARK

New acid plant catalyst

At the Sulphur 2017 conference in Atlanta, Haldor Topsoe launched a new highly active SO_2 oxidation catalyst for sulphuric acid plants. *VK711 LEAP5*™ aims to achieve compliance with emissions standards without compromising on performance or venturing into costly revamps or tail gas treatment with scrubbers. The catalyst has been optimised for higher activity at lower temperatures, which allows for reduced operating costs as well as the choice of maintaining the same production level with lower emissions or increasing productivity without increasing emissions. The new catalyst allows operation from temperatures as low as 370°C and is an obvious choice for single absorption acid plants. Other applications include oleum production and more efficient desulphurisation of flue gases.

Topsoe's new VK711 LEAP5 *sulphuric acid catalyst.*

CHINA

Pollution crisis leads to smelter shutdowns

Tongling Nonferrous Metals Group Co., China's second largest copper smelter, says that it has idled 20-30% of its 800,000 t/a of copper smelter capacity at Tongling in Anhui province, due to government-mandated curbs intended to ease pollution during the winter. As yet there is no projected re-start date for the smelter capacity. Anhui province's government has asked polluting industries such as steel, cement, and non-ferrous metals to cut capacity by more than 30% percent this winter.

The move has boosted copper prices worldwide and is expected to have a significant impact on the current annual round of negotiations between Chinese smelters and overseas mining firms over ore processing fees. China is the biggest producer and consumer of refined copper and relies on some of the world's largest miners for its copper concentrate. China's smelters say they are being forced to upgrade facilities to comply with environmental rules, and charges should reflect their higher costs. However, miners point to a projected market deficit of 50,000 t/a of copper for 2017 and risks of supply cuts due to strikes in important mining companies.

Elsewhere, China's Hongyue North Copper has begun production from a new smelting project in northeastern Liaoning province, according to the company. The smelter, which took 22 months to build, will have an annual capacity of 150,000 t/a of refined copper, 5 t/a of gold and 300 t/a of silver.

AUSTRALIA

BHP defers Olympic Dam plans

BHP Billiton has deferred plans to increase output from its giant Olympic Dam copper mine in Australia to 450,000 t/a, opting instead for a less ambitious expansion project. The world's biggest mining company says that its preferred development option is now a $2.1bn plan that will see output rise from an estimated 150,000 t/a this year to 330,000 t/a by 2023 via a brownfield expansion to handle high grade ore from the southern area of the mine. The BHP's board will be asked to approve the project in mid-2020, in anticipation of a deficit emerging in the global copper market.

SYRIA

SSP plant re-starts

The Syrian Arab News Agency (SANA) reports that the General Fertilizer Company's single superphosphate (SSP) fertilizer plant in the (GFC) at Homs has resumed production in December 2017 after two years of downtime. The plant has a capacity of 350 t/d (115,000 t/a). Syria's Industry Minister Ahmad al-Hamo reportedly said during a tour of the plant that resuming production in the plant came after securing the raw materials needed for it, hailing the efforts of all employees of the company and their insistence to stay in their company to provide the needs of the farmers.

BRAZIL

Yara to buy Cubatao

Yara says that it has entered into an agreement to acquire the Vale Cubatao Fertilizantes complex in Brazil from Vale for $255 million. It forms part of Yara's plans to establish itself as a nitrogen producer in Brazil, complementing its existing distribution position. Cubatão is a nitrogen and phosphate complex with an annual production capacity of approximately 200,000 t/a of ammonia, 600,000 t/a of nitrates and 980,000 t/a of phosphate fertilizer. It also includes a 1,100 t/d sulphur-burning sulphuric acid plant. Sulphur and other raw materials are supplied via a nearby import terminal which is not part of the transaction. Closing is expected to take place in 2H 2018.

Yara says it expects to make upgrading investments of approximately $80 million up to 2020 in order to realize annual synergies of $25 million through a combination of cost, asset and product portfolio optimisations.

"This deal is an important step towards establishing a more complete position in Brazil, strengthening our position as a long-term competitive industry player, committed to developing and investing in Brazilian agriculture and industry," said Svein Tore Holsether, president and CEO of Yara.

Ukrainian rail cars destined for Turkmenistan.

UKRAINE

Sulphur rail cars for Turkmenistan

Ukraine's PJSC Azovobshemash has obtained an order to manufacture 20 tank new generation railway tank carriers for the transportation of sulphuric acid for the Turkmenabat Chemical Plant in Turkmenistan, replacing old units which have reached the end of their economic life. Azovobshemash, based in Mariupol, says that the tank cars will have "improved technical and economic features". The contract requires an accelerated build time for the new cars. It forms part of an order of 1,500 freight cars which Ukraine is building for Turkmenistan, agreed at a recent meeting between Turkmenistan deputy prime minister Satlik Satlikov and Ukrainian deputy prime minister Gennady Zubko.

INDIA

Construction to begin soon on OCP joint venture NPK plant

Morocco's minister for logistics and transport, Abdelkar Amara says that construction of the new joint venture Kribhco-OCP NPK plant in Andhra Pradesh is expected to commence in the next few months. The 50-50 joint venture plant will be built at the port of Krishnapatnam in Andhra Pradesh, and will have a capacity of 1.2 million t/a. Front end engineering and design work on the $230 million facility is under way, and construction is expected to begin by July 2018. The plant will import phosphate rock from OCP in Morocco to make the compound fertilizer.

CHILE

Strike at Enami copper smelter

A strike at the state-owned Hernan Videla Lira smelter at Paipote in northern Chile halted operations from mid-December to early January, according to state mining development agency Enami. Members of the No 2 Workers Union at the metallurgical complex began their protest after talks over a new collective contract ended without agreement. The smelter, in the copper-rich Atacama region, produced 84,500 tonnes of copper anode in 2016.

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People

Arianne Phosphate has named **Dominique Bouchard** as Executive Chairman of its Board. Mr. Bouchard has been a member of Arianne's Board since 2013 and his new position follows his previous role as Arianne's Executive Vice Chairman. He has also been Vice Rector of Resources between 2014 and 2017 at the Université du Québec in Chicoutimi. Prior to that he served for 33 years at Alcan and Rio Tinto, most recently as President of Rio Tinto Quebec Iron & Titanium until his retirement in May 2013. He also held the position of Vice President Primary Metal within Rio Tinto Alcan from March 2005 to February 2010, and was responsible for operations and implementation of strategy development for Primary Metal Saguenay-Lac-Saint-Jean. Bouchard holds a Master's degree in Management from McGill University, and is also a graduate of the Institute of Corporate Directors.

"I believe I am well suited for my new role as Executive Chairman," said Dominique Bouchard. "As a native to the Saguenay region, I have a fundamental understanding of how important the Lac à Paul project is, and, the benefit it will bring to the community. Having spent many years in Québec, both in a personal and professional capacity, it has allowed me to build a strong network of contacts that I will draw on to help get this project done. Further, I look forward to continuing my strong working relationship with Brian, our CEO. We have a strong understanding of each other's strengths and responsibilities and, we work well as a team."

"On behalf of the Board, the Management and the entire Arianne team, I want to welcome Dominique to his new position," said Brian Ostroff, CEO of Arianne Phosphate. "I have had the opportunity to work with Dominique over the years and think he is very well suited to his new position; I look forward to advancing our project together. Having been on the Board for several years Dominique will bring a high level of continuity and can hit the ground running. Lastly, on a personal note, I have enjoyed working closely with Dominique in the past and look forward to his more active role as Executive Chairman."

Chatham Rock Phosphate Limited says that **Justin Cochrane** has retired as a director, due to increased responsibility and board commitments at Cobalt 27 Capital Corp., which have required him to step down from his current duties on the board of Chatham Rock Phosphate. The company said in a statement that "we regret his loss, as Justin played a key role in assisting the successful transition of Antipodes Gold into Chatham Rock Phosphate, but we congratulate him on his Cobalt 27 promotion."

The board of KazMunayGas has appointed **Ospanbek Alseitov** as general director of Pavlodar Petrochemical Plant LLP.

Kuwait has appointed **Bakheet Al-Rashidi**, head of the country's international refining unit, as its oil minister. Al-Rashidi, president and chief executive officer of Kuwait Petroleum International, joins the cabinet as part of a change in the Gulf country's government, according to a royal decree published on the official news agency KUNA. He replaces Issam Almarzooq, who held the position since December 2016. KPI is a unit of state energy producer Kuwait Petroleum Corp. Al-Rashidi has spent most of his career with Kuwait National Petroleum Co., KPC's domestic downstream arm, heading functions ranging from operational planning to technical services and corporate planning. From 2007 to 2013, he was KNPC's deputy chairman and deputy managing director for planning and local marketing. He has served on the board of Kuwait Oil Co., KPC's upstream arm, and as chairman and managing director of a local joint venture, Kuwait Aromatics. He graduated from Alexandria University in Egypt with a degree in chemical engineering, according to KPI's website.

Calendar 2018

FEBRUARY

25-28

Laurance Reid Annual Gas Conditioning Conference. NORMAN, Oklahoma, USA
Contact: Tamara Powell, Program Director
Tel: +1 405-325-2891
Email: tsutteer@ou.edu

MARCH

5-9

Brimstone Sulphur Recovery Training Course, HOUSTON, Texas, USA
Contact: Brimstone-STS
Tel: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

11-13

AFPM Annual Meeting, NEW ORLEANS, Louisiana, USA
Contact: American Fuel and Petrochemical Manufacturers (AFPM), 1667 K Street, NW, Suite 700, Washington, DC 20006, USA
Tel: +1 202 457 0480.
Email: meetings@afpm.org
Web: www.afpm.org

12-14

Phosphates 2017, MARRAKESH, Morocco
Contact: CRU Events
Tel: +44 20 7903 2167
Email: conferences@crugroup.com

18-21

Middle East Sulphur, ABU DHABI, UAE
Contact: CRU Events
Tel: +44 20 7903 2167
Email: conferences@crugroup.com

19-23

Sulphur Experts Technical Training Course, KUALA LUMPUR, Malaysia
Contact: Sulphur Experts Training Coordinator
Tel: + 1 281 336 0848
Email: Seminars@SulphurExperts.com

26-30

SOGAT 2018, ABU DHABI, UAE
Contact: Dr Nick Coles, Dome Exhibitions
Tel: +971 2 674 4040
Email: nick@domeexhibitions.com

APRIL

30 - MAY 4

Sulphur Experts Technical Training Course, KEMAH, Texas, USA
Contact: Sulphur Experts Training Coordinator
Tel: + 1 281 336 0848
Email: Seminars@SulphurExperts.com

JUNE

8-9

42nd AIChE Annual Clearwater Conference 2017, CLEARWATER, Florida
Contact: Perry Alonso, AIChE Central Florida Section
Email: vice-chair@aiche-cf.org

18-20

86th IFA Annual Conference, BERLIN, Germany
Contact: IFA Conference Service, 28 rue Marbeuf, 75008 Paris, France.
Tel: +33 1 53 93 05 00
Email: ifa@fertilizer.org

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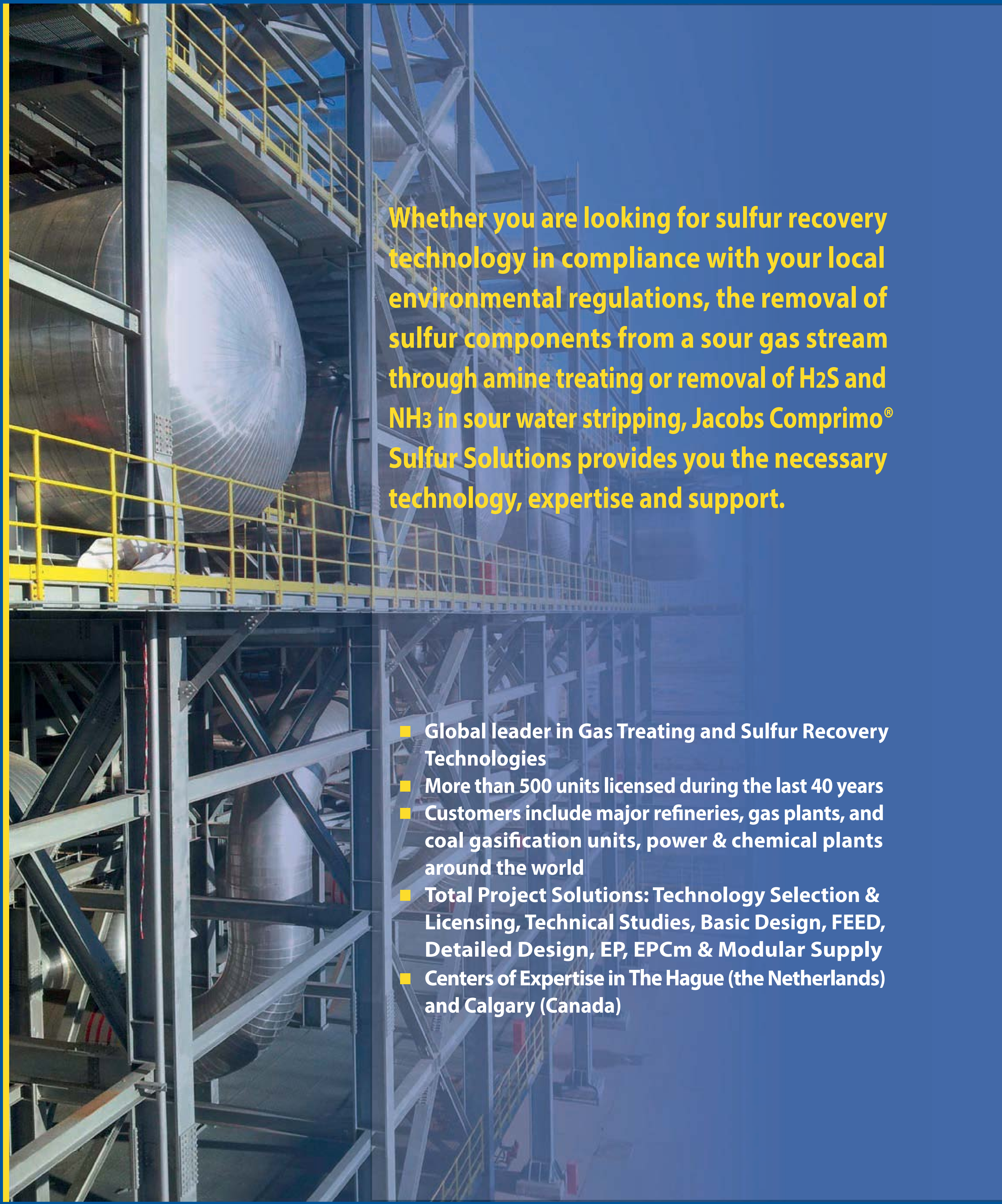

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Sulphur's sea change

The impact of new International Maritime Organisation (IMO) rules on sulphur content of shipping fuels and sulphur dioxide emissions from shipping are proving to be a headache for shippers, refiners and potentially the entire sulphur industry.

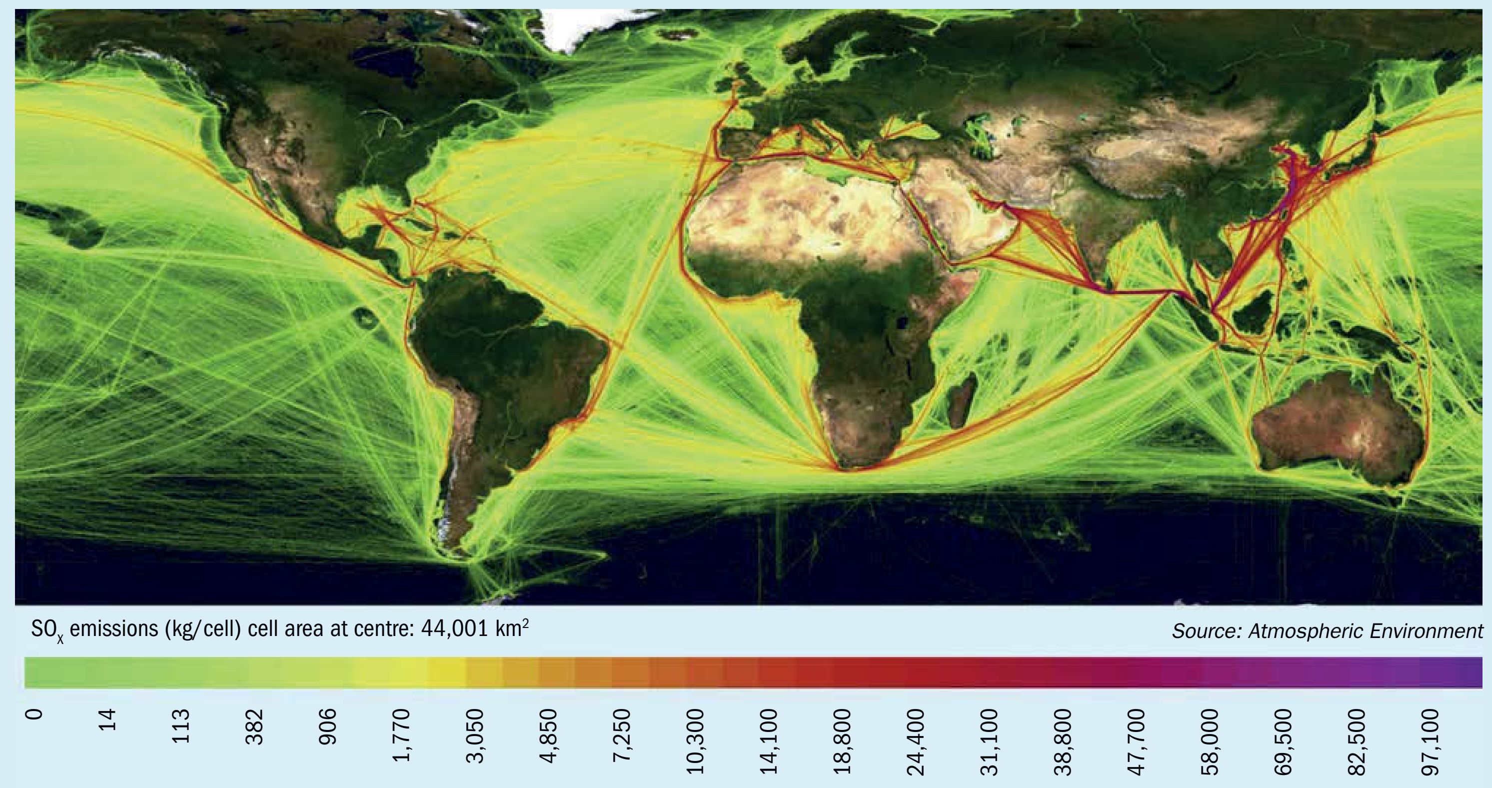
From January 1st 2020, the maximum permissible sulphur content of marine bunker fuels will be 0.5% by weight. This target has been set by the International Maritime Organisation (IMO) in order to reduce sulphur dioxide emissions from ships and associated health risks for people living in coastal areas. However, with only two years to go before the implementation deadline, the number of ships which have converted to scrubbing technology and the number of refiners capable of supplying low sulphur bunker fuels remains far short of what will be required to avoid a major price shock for shipping companies.

The issue

The issue that the regulation is trying to address is the effects to human health caused by airborne emissions of sulphur dioxide (and particulate matter). Sulphur dioxide has long been known to be a threat to human health, but medical studies have begun to find that long term exposure even to relatively low levels has significant effects on human health, especially among those most susceptible (children, asthmatics etc). In 2005 the World Health Organisation lowered its long term exposure guidelines for SO_2 from 125 to 20 µg/m³. The knock-on effect has been a tightening of sulphur content restrictions in all fuels, and in 2008 the IMO adopted Annex VI to its MARPOL maritime pollution guidelines which set a pathway towards a gradual phase-out of sulphur in marine bunker fuels. Designated emission control areas are already in force (since 2015) with a 0.1% limit on sulphur content of fuels burned in those areas, mainly off the east and west coasts of North America, and the North and Baltic Seas around Europe. However, while 70% of sulphur emissions from shipping occur within 200 miles of coastlines, this still left most of the world's coastlines not protected by emission control areas. The other prong of Annex VI is a stepwise reduction in sulphur content of fuels burned anywhere at sea. A global reduction to 3.5% in 2012 had no major effect, as most bunker fuels were already below that level. However, the drop of that cap to 0.5% is expected to have a much greater effect.

The original Annex VI agreement provided for the 0.5% global cap to be introduced in either 2020 or 2025, depending on the ability of the shipping and refining industries to comply with the regulation. However, in 2016 the IMO decided to proceed with the introduction of the cap in 2020 regardless. The basis of the IMO decision to proceed with the reduction in 2020 rather than delaying it to 2025 was a report by Finland submitted to the IMO's Marine Environmental Protection Committee (MEPC) in 2016[1]. The study found that a reduction from 3.5% to 0.5% sulphur in maritime fuels outside of established emissions control areas (ECAs) would reduce SO_2 emissions by 8.5-9.0 million tonnes between

Fig. 1: SO_x emissions from shipping



2020 and 2025, leading to an approximate 77% reduction in global SO_2 emissions from international shipping. Emissions of particulate matter would be reduced by 0.76-0.81 million t/a, amounting to a 50% reduction. The effect of these lowered emissions would be a significant reduction in exposure to harmful air pollutants, especially in populated coastal areas, and would prevent more than 100,000 premature deaths per year (the low estimate was around 40,000 deaths per year, the high estimate 175,000 deaths). It was therefore estimated that over the five-year period a total of 570,000 premature deaths will be avoided.

As Figure 1 shows, the effects are most pronounced in the Mediterranean and Red Seas, Indian Ocean, Arabian Gulf, and the coasts of Southeast and East Asia (the seas around Europe and North America already have much stricter fuel sulphur standards due to the existing ECAs). Consequently, more than 90% of these health benefits are expected to take place in the Asia-Pacific region, Africa and Latin America.

Amelioration options

Two main options exist for complying with the new regulations; switching to use of low sulphur fuels, or the fitting of amelioration technology to ships – these are generally scrubbers which take the exhaust air from the engines and remove the SOx, NOx and particulates from it before the exhaust is released to air. However, within these apparently relatively simple options lurk various issues and complications.

Scrubbers

So-called secondary amelioration is possible by retrofitting an engine exhaust scrubbing system to a vessel, which can remove around 99% of SOx emissions. The two main options are 'wet' and 'dry' scrubbing. Dry scrubbing uses granules of calcium hydroxide in a filter system. The $Ca(OH)_2$ reacts with SO_2 to form gypsum, $CaSO_4$. As it is a closed system there is no discharge to deal with, and power requirements are relatively low. However, there is a considerable weight penalty with such a scrubbing system, the difficulties of handling a potentially hazardous substance, and the calcium hydroxide is consumed in relatively large quantities – a 20MW engine used in a large vessel would consume 19 tonnes per day of hydroxide. Only one dry scrubber is currently offered for marine use.

Wet scrubbing systems fall into two types; closed and open loop systems. Open loop systems use seawater, dissolving SOx in the water to form sulphites and bisulphites, and allowing dissolved carbonates and bicarbonates in the seawater to neutralise them to sulphates. Closed loop systems use fresh water and a tank of sodium hydroxide solution, producing sodium sulphate. A major issue with wet scrubbing systems however is discharge of waste water. Open loop systems require continuous discharge of often acidic waste water, while closed loop systems can operate in zero discharge mode for a certain period, but require bleed off after that. Regulators have begun to become concerned about acidification of water, especially around coasts or in freshwater river systems, and it is possible that more regulation may follow in this area. Closed loop systems are also of course more expensive than open loop systems, and both must deal with corrosion from acidified water and exhaust back pressure into the engine.

As well as the capital and operating expenditure of scrubbing systems, and concerns over acid discharges, another of the issues for shipowners is the potential for investing in 'over control' of SO_2 emissions – even for a conventional 3.5% sulphur HSFO, a scrubbing system will typically reduce emissions to below those for an equivalent 0.1% sulphur fuel, as specified in emissions control areas – this means that part of the decision on whether to install a scrubbing system depends on how long a vessel will spend in ECAs. The current reckoning, according to the IMO, is that a vessel at present needs to spend around 4,500-5,000 hours per year travelling in an ECA for the installation of scrubbers to be worthwhile, and that only for open loop scrubbers. At present, closed loop scrubbers do not ever seem to be the cheaper option (compared to switching to a low sulphur distillate fuel)[4].

The upshot of the costs and uncertainties of scrubbing systems is that adoption rates have been quite low so far; around 28% of the cruise ship industry has installed exhaust scrubbing systems, but uptake among cargo ships has been far lower. There are an estimated 60,000 vessels covered by the IMO legislation, of which only 400 have so far installed scrubbers or placed firm orders to do so, mainly those operating primarily in ECAs. There is also a potential bottleneck in terms of the number of suppliers and dry dock facilities capable of installing these systems, estimated at about 1,200-1,800 vessels per year. Shell has estimated that the number of scrubber equipped ships will be no higher than 1,500 by 2020[3]. This will be skewed towards larger vessels with heavier fuel consumption (30% of vessels represent 70% of bunker fuel consumption), and so while this is only 2.5% of the merchant fleet, it could represent 5-8% of fuel demand. Nevertheless, it is clear that scrubbers are not going to significantly impact demand for lower sulphur marine fuels in the short to medium term.

Fuel switching

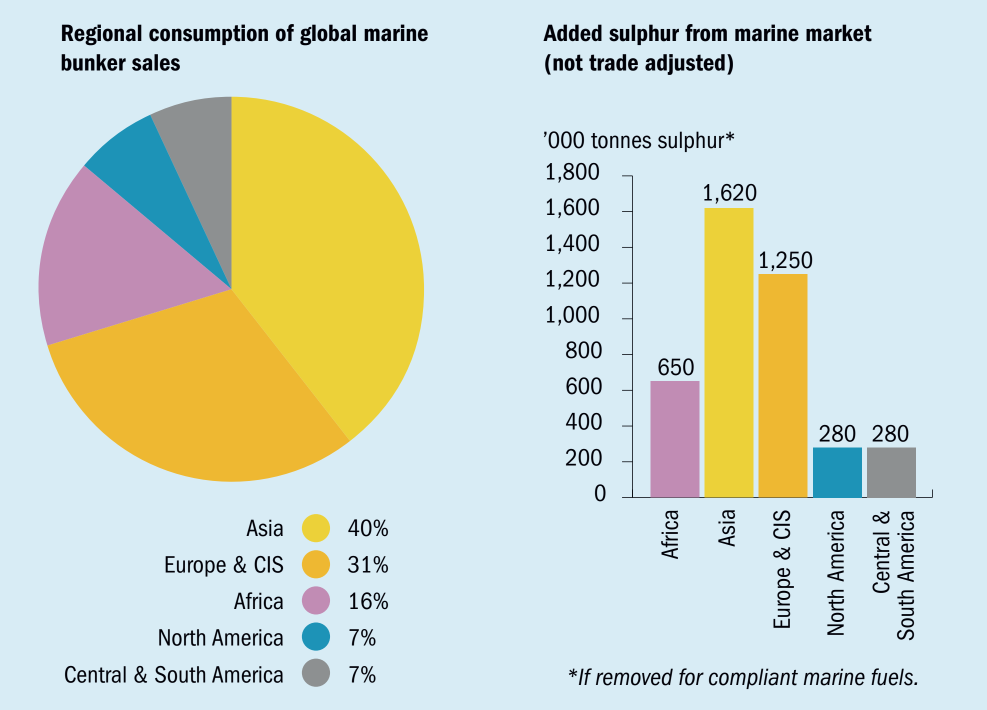
While the focus is on a switch from high sulphur fuel oil (HSFO) to marine gasoil (MGO) – effectively diesel – other low sulphur fuels are available. The most widely touted alternative is liquefied natural gas (LNG). Other alternatives include methanol. Once again, however, take-up has been slow, and mainly concerns new vessels. There are about a dozen methanol vessels and around 200 LNG ships in service or on order. Again the cruise sector has been a faster adopter, with around 15% of ships now running on alternative fuels, but again, with less than 0.5% of the shipping fleet so far operating on alternative fuels, it is a safe bet that by 2020 take-up will not have had much impact on demand for low sulphur bunker fuels. Longer term, some projections put the market share for LNG-fuelled at up to 13% of the commercial ship market by 2025. However, the issue then is that it may restrict the number of ports available to a vessel, and so may make most sense for ferries or container ships running predictable routes or to large ports that are likely to have the fuel infrastructure to support it.

Non-compliance

The other, and perhaps final option for ship owners, is of course non-compliance with the regulation. Estimates of this vary, but figures of up to 30% have been suggested, depending on enforcement and fines, with possible incentives to deliberately divert ships to 'non-available' ports where there is no low sulphur fuel sold. The model for this is the introduction of the 2015 emission control area limits. At present, fines and enforcement mechanisms are down to the individual member states of the IMO. This means that they can be extremely variable. A study by Maersk reported that in the 2015 European ECAs, recorded non-compliance rates in port inspections conducted were 3% in the Baltic Sea and 9% in the North Sea, but that only 30% of violations were sanctioned. In some

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46
47 48 49 50 51 52 53 54 55 56

Fig. 2: **Potential recovered sulphur from marine fuels**



Source: IMO

countries, fines were as low as $1,500, compared to potential savings of up to $100,000 per trip, per ship, from using non-compliant fuel. There were very few detentions and very few cases of legal action[6].

Refiners

All of this, then, passes the burden of compliance back onto the refining industry, who in 2020 are going to be expected to have to produce potentially as much as 3 million bbl/d of extra low sulphur fuel for shipping. Shell estimates that although there will be 1.5 million bbl/d of coker capacity installed by 2020, there will still be around 1.5 million bbl/d of excess HSFO and too little marine fuel oil/gas oil (MFO/MGO) by that time. The IMO has tried to minimise expectations of disruption; it conducted a study in 2016[2] which concluded that there is enough capacity to provide compliant fuel in 2020, albeit with a 5.3% tightening of the market for marine distillates – within tolerances and spare capacity according to the IMO. However, one of the study's assumptions was that there would be much wider take-up of scrubbing technology – it reckoned on 4,000 ships being equipped by January 1st 2020 (although its figures for LNG and alternative fuels are roughly correct at around 200). As explained by one of the report's authors, James Corbett, to the sulphur industry at the Sulphur 2017 conference in November[4], there will nevertheless be regional imbalances which will need to be addressed either by transport of fuel from one region to another or changing of ships' bunkering patterns.

In order to meet the demand for extra marine distillates, refiners are going to need to make substantial investment in upgrading fuel oil residues to gasoil grades via secondary units such as crackers,

visbreakers and cokers, but this may be very location specific according to where the best returns are likely to be. There will also no doubt be an attempt to limit residue production by changing to a sweeter crude slate, although in the context of 2020 this may well increase the price spread between sweet and sour crude grades to much higher levels than at present. Other options include residue destruction – an expensive prospect – or desulphurisation of residual fuel oil and blending with low sulphur gasoils – a more expensive option than simply upgrading, and at present not one many refiners have gone for.

Supply of hydrogen may also be an issue, and all of this will boost refinery CO_2 budgets in areas which penalise them for such, such as Europe.

No doubt a variety of blends will be available to try and meet the 0.5% limit. Many Far Eastern crudes have less than 0.1% sulphur in their vacuum gasoil fraction, allowing it to be blended into the residue. But trialling new blends also requires research and development effort if there are not to be nasty surprises for customers, and it has been suggested that a timeline of 4 years may not be excessive.

Overall, margins for simple refineries that turn a significant share of their crude run into HSFO will be constrained, but complex refineries may find themselves better placed to take advantage of the new situation.

Additional sulphur

HSFO has traditionally been a sink for refinery sulphur, and at a limit of 3.5% this means that there are millions of tonnes of sulphur potentially to be removed. What effect might this have on the sulphur market? The IMO calculates that if all high sulphur fuel oil had its sulphur removed down to the 0.5% cap, this would represent an additional 4.1 million t/a of sulphur recovered by refineries, or approximately a 15% increase in the global sulphur supply from refining, which totals around 28 million t/a. This would be geographically distributed as shown in Figure 2. This extra sulphur is likely to make little difference the North American market, where it represents only an additional 3%, but in Europe and South America this represents a 25-30% increase in refinery sulphur, and in Africa a 200% increase. Of course, not all residue is likely to be upgraded, and as noted the presence of scrubbers and probably considerable non-compliance will likely make a significant dent in the actual volume of sulphur ultimately recovered. Still, in a world facing a surplus of sulphur over the next few years, these extra volumes are only likely to make the pressure to store sulphur or find other uses for it all the more intense. ■

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Krylatskoye Bridge, Russia – resurfaced by Gazprom with sulphur asphalt.

PHOTO: GAZPROM

A new lease of life for sulphur concrete?

Sulphur's use as a binding agent to produce tough, chemical-resistant concrete has a long history, but little commercial success to show for it. But new concerns about conventional concrete's CO_2 output and worries about sulphur surpluses in some regions are leading to renewed interest.

The use of sulphur as a strengthening agent in materials dates back centuries. Sulphur's ability to link between polymer strands or other components of a substance has seen it used in rubber (vulcanisation), and more recently in sulphur polymers (see *Sulphur 366*)[1]. In the area of construction, US patents from 1844 and 1859 describe the use of sulphur in an improved cement, and in 1900 another described a sulphur composition "suitable for roofing, conduits, pavements, ornamental figures and the coating of steel ship hulls"[2]. In 1920, a sulphur sand mortar was used in a sewer pipe carrying acidic waste to resist acidic attack. Experiments continued in the 1920s and 30s on cements and concretes made from sulphur coke compositions and sulphur aggregate compositions for use in flooring and acid-resistant tanks and pipes[3].

However, the most recent phase of sulphur concrete research can be dated to Canada in the early 1970s, when a growing surplus of sulphur from Canadian sour gas production led to a search for new uses for the material. The Canadian federal government, together with the Alberta provincial government and several sulphur-producing companies established the Sulphur Development Institute of Canada (SUDIC) in Calgary to examine potential new markets for sulphur. Alan Vroom, formerly of the Canadian National Research Council, wrote several papers describing sulphur's potential use in concrete and similar materials, and by 1974 had conducted work with the University of Calgary's Department of Civil Engineering to prove the concept.

The concept was to replace the water and cement in Portland cement concrete with sulphur. Portland cement concrete is a mixture of around 45% aggregate, 25% sand, 12% cement and 15% water. The equivalent sulphur material, which became known as *Sulphurcrete*, was 47% coarse aggregate, 27% sand and 25% sulphur. It could be melted together above the freezing point of sulphur, and then left to set. The lack of water meant that it did not suffer from shrinkage in the same way that conventional concrete did, although creep was, as with normal concrete, still a potential issue. It was also found to achieve its maximum strength much more quickly than conventional concrete. Finally, it was relatively cheap; at the time, sulphur was being produced at sour gas plants in Alberta for about C$12.00/tonne, and the cost of Portland cement meant that sulphur concrete was actually cheaper than Portland cement concrete (estimated in 1974 at C$10.35/cubic yard for sulphur concrete, compared to C$14.20/cubic yard for Portland cement concrete)[3].

Sulphurcrete

There were however still key issues with sulphur concrete which prevented its take-up as an engineering material. Adding only pure sulphur to the concrete led to excellent initial strength, but it was found to eventually lead to material failure as the unmodifed sulphur converted from its 'plastic', monoclinic crystal form to a more brittle orthorhombic crystal structure with poor strength characteristics. The sulphur also underwent a volume and density change as it changed from one phase to another, losing 8% of its volume in the process and hence stressing the concrete, producing cracks. The concrete also could not bear high temperatures – above 115°C, the sulphur in the mix simply melted and the concrete dissolved back into its components, although this could also be sold as a benefit, allowing the material to be recycled. Finally, the sulphur concrete produced was brittle and subject to cracking when exposed to multiple freeze-thaw cycles.

The solution to this was to add a sulphur polymer modifier to try and preserve the monoclinic sulphur form by encouraging the formation of polysulphide chains. Considerable testing of materials isolated

1 47 2 48 3 49 4 50 5 51 6 52 7 53 8 54 9 55 10 56 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46

the best additive for this, dicyclopentadiene (DCPD). Called '*SRX*' as a trademarked additive, the sulphur concrete with modifier was licensed by its developer, Alan Vroom, who founded his own company, Starcrete, based in Toronto, to sell it as a material also known as *Starcrete*.[4] At the same time, the rights to the technology in North and South America and the Pacific Rim were also sold to Canadian mining company Cominco, based in Vancouver. Cominco also manufactured DCPD, and hence were able to supply what they called *Sulphurcrete*, with its proprietary *SRX* modifier.

The new material not only preserved the original *Sulphurcrete* formulation's excellent initial strength (achieved in minutes or hours as compared to days for conventional concrete), but also led to it being stronger overall than Portland cement concrete. However, this also made tolerances finer when pouring the concrete – smooth pouring must be accomplished quickly before the material begins to solidify. Application or removal of heat could however alter this curing time. It retained the issue of potentially re-melting at high temperatures (>120C), but the low thermal conductivity of the material protected it from short-term temperature excursions.

Sulphurcrete's new composition was a mix of coarse and fine aggregates (total of 82%), 5% mineral filler, 11.5% sulphur, and 1.2% *SRX* modifier – lower volumes of sulphur, it had been discovered, also led to lower shrinkage cavities and less internal stress. It was used in industrial flooring and corrosion resistant applications, and its ability to be poured in low temperatures made it especially suitable for use in pipeline applications in freezing Canadian winters (for supports or weights).

SulfCrete

The problem with *Sulphurcrete* was that the *SRX* modifier considerably increased the cost of the material – DCPD currently retails for around $2,000/tonne in bulk. The price of sulphur, which had spent most of the 1970s in the $30-40/t region f.o.b. Vancouver also rose during the 1980s as it became increasingly used in phosphate fertilizers, averaging around $100/t across the decade. All of this served to change the economics of the product considerably. It consequently found use in some niche areas, but did not achieve mass commercialisation.

However, a new formulation has since come along due to work conducted in the US via the Brookhaven National Laboratory (BNL) on Long Island, New York. Nuclear engineer Paul D Kalb at BNL was trying to solve a problem with the long-term disposal of radioactive waste by encasing it in concrete. However, the issues was that the radioactive waste (mainly ash) did not mix well with Portland cement, leading to porosity and leaching of the waste from the concrete. To try to correct this, Kalb's team began working with sulphur concrete and examining ways that it might encapsulate the ash. He developed a sulphur polymer solidification process which was subsequently patented by BNL, and which inadvertently seems to have solved one of the problems with sulphur concrete by removing the need for DCPD as a modifier. The new process involves pre-treatment of the filler materials (in this case fly ash and quartz aggregate) with a light catalytic cracking oil that is a by-product of the refining industry, followed by processing with elemental sulphur to form the polymerised sulphur mortar. The mortar mix is typically 54% sand, 18% ash, 26% sulphur and 2% organic modifier[5]. BNL describes this as Stabilised Sulphur Binder using Activated Fillers (SSBAF).

In 2012 a commercialisation agreement was signed between BNL's commercial arm Brookhaven Science Associates LLC and Green SulfCrete, a technology start-up company based near to BNL. Since then, the partner company has changed its name from Green SulfCrete to just SulfCrete and has entered into a 20 year license agreement with Brookhaven for the technology. SulfCrete has accrued $2 million in funding and is in negotiations for a further $5 million, in conjunction with their partner, local concrete manufacturer Roman Stone Construction Company in Bay Shore, Long Island, to build a commercial-scale production plant this year.

The company describes its new product, *SulfCrete*, as not only a stronger but also a more environmentally friendly alternative to traditional concrete. Since the 1970s, the concrete industry has moved on, and one of the major concerns to day is the amount of energy required to melt limestone for cement. Temperatures of over 1,450C are required, generating high levels of carbon dioxide – some 5-10% of all global greenhouse gas emissions are estimated to come from come from cement production for concrete, which generates 0.6-0.95 tonnes of CO_2 for every tonne of concrete, depending on the efficiency of the cement kiln. Large volumes of water are also required, which can be increasingly problematic in some areas. *SulfCrete* however generates 93% less CO_2 than conventional concrete, and the sulphur polymer mortar is more than three times stronger than traditional Portland cement mortar. SulfCrete also say that it is also now 33% cheaper than conventional Portland cement concrete in the US, assuming sulphur at $100/tonne, due to the lower cost of the additive ($100/t compared to $2,000/t for DCPD).

In addition to SulfCrete, BNL has also been involved in working with the authorities in Kazakhstan on the potential use of sulphur concrete there – Kazakhstan has been worried about potential stockpiles of sulphur from oil and gas processing activities, and several years ago fined the TengizChevroil consortium considerable amounts of money over alleged problems relating to fugitive sulphur dust from sulphur blocks and forced the sale of TCO's sulphur stockpile.

Thiocrete

Independently of Cominco's *Sulphurcrete*, Shell Canada – a major producer of sulphur – began its own work on developing sulphur concrete and asphalt. Shell launched its Sulphur Extended Asphalt product (now *Thiopave*) in 2003, and its concrete binder mix soon after in 2005. Shell's *Thiocrete* is supplied in liquid or pellet form as a combined sulphur and modifier mixture. It can then be mixed with aggregate at 135C and poured into moulds. *Thiocrete* has been trialled in Canada, at Shell's Waterton gas plant in Alberta, and Shell opened a research centre in Qatar in 2005 and has also been trialling various concrete mixes there since 2008. From 2008-2011, panels of *Thiocrete* were also trialled at Ijmuiden in the Netherlands in the tidal zone of the breakwater, and were found to suffer far less wear and tear than conventional concrete. Shell has also worked with other companies, such as Belgian concrete manufacturer DeBonte International, the fruits of which have been installations of sulphur concrete tram and railway sleepers in 2013 and 2014, and the *Thiotube* range of sewer pipes, connectors and manhole covers in 2015.

More information on Shell's value-added sulphur technologies can be found in Sulphur 347[6], but currently Shell is prioritising its sulphur enhanced fertilizers rather than its asphalt and concrete technologies.

Serobeton

While sulphur concrete has so far remained a niche market in most of the world, there has been considerable interest in the material in Russia, particularly from Russia's main sulphur producer, Gazprom, as a potential outlet for its own sulphur production. Gazprom's worry is that it will have an increasing sulphur surplus over the coming years, and Gazprom chairman Alexei Miller has gone so far as to say that the inability to sell a sulphur surplus from the Astrakhan and Orenburg gas plants is one of the key constraints in lifting production there (original plans from the 1970s for sour gas production at Astrakhan could have seen up to 16 million t/a of sulphur production).

The company began with developing a sulphur asphalt; in 1998 Gazprom VNIIGAZ began investigating the possibility of using sulphur asphalt in road construction and in late 2002 it was used to repair the road surface of a bridge in Krylatskoye. In June 2010, a total of 558 tonnes of sulphur asphalt were laid on a 50km stretch of the Moscow Ring Road. The Russian Ministry of Transport is now partnering Gazprom in a number of pilot projects for using sulphur asphalt in regions with different temperature conditions.

However, the amount of sulphur in sulphur asphalt is relatively low (only 1-2%), and hence the potential for this market within Russia could only account for a maximum 200-300,000 tonnes of sulphur per year, according to Gazprom. Consequently, the company has also begun to look more seriously at sulphur concrete, where sulphur content in the binder is up to 90%, and the potential market in Russia could be as high as 5 million t/a, via its subsidiary Gazprom Sero (Sulphur).

Gazprom has been conducting a programme of research and development and developing a regulatory framework for the new materials, and is developing a pilot production plant at Astrakhan for its new sulphur modified binder, a mix of 93-98% sulphur and 2-7% dicyclopentadiene. This is then used in both its *Seroasphalt* sulphur modified asphalt and *Serobeton* sulphur concrete. Gazprom suggests that it could be manufacturing 120,000 t/a of the sulphur binder in a few years' time, along with sulphur concrete and asphalt mixes, road slabs for surfacing temporary and permanent roads, square reinforcing piles, and gas pipeline wrap-around concrete weights.

In 2016 Russia produced 6.1 million t/a of sulphur, exporting 3.8 million t/a of this. Gazprom was responsible for nearly 85% of these exports. Gazprom says that it further expects Russian sulphur production to rise to 7.5 million t/a by 2020.

Tiocomposite

Gazprom is not the only Russian company involved in sulphur concrete research. In 2014, a new company was established in Russia's Tatarstan region, a collaboration between the Nanotechnology Centre of the Republic of Tatarstan and a Kazan-based van manufacturer. Tiocomposite LLC's managing director, Evgeny Khramov, was a physicist who had worked on sulphur copolymers since 2004 at Kazan Federal University, initially in rubbers, and then in the production of building materials. The company has built a pilot plant for the manufacture of sulphur concrete products and conducted trials which led to the production of large-scale products made from sulphur concrete. Kazakhstan has been interested in the development of sulphur concrete based sewage and water supply systems, and Tiocomposite has also been involved in laying a 350 metre experimental stretch of road using slabs of sulphur concrete. The company has a mobile plant for production of sulphur concrete based on four 16 metre truck platforms, which can produce a total of 20 t/h, and says that it is encouraged by the interest of large oil and gas companies in Russia in looking for an outlet for their sulphur, especially Lukoil, Gazprom and RAO. Norilsk Nickel, now that it is producing sulphur from metallurgical offgases, is also looking for an outlet for its sulphur production.

Laying sulphur concrete roadway slabs in Russia.

PHOTO: TIOCOMPOSITE

A concrete future?

The cost of the DCPD modifier and the highly variable cost of sulphur itself have generally been the barriers to the more widespread adoption of sulphur concrete. The new Sulf-Crete formulation may potentially overcome this, and it will be interesting to see where this leads over the next few years. Sulphur concrete has always had a winning story in terms of its strength, durability and lifespan and its low permeability and resistance to chemical attack. Its low carbon intensity compared to conventional concrete is now also becoming an increasingly important factor, although any benefit from this would require regulatory bias in its favour in terms of either some form of carbon pricing or its selection as a 'best available technique' in favoured applications. But finally, if the cost hurdle can also be overcome, then its future might be all the brighter. In the meantime, the greatest interest comes, as it did in the 1970s, in regions where there is a looming sulphur surplus, now Central Asia, especially Kazakhstan and Russia.

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Sulphur 2017

Highlights of the Sulphur 2017 conference, held in Atlanta, Georgia in November.

PHOTO: SEAN PAVONE/SHUTTERSTOCK.COM

Main pic: Skyline and reflections of midtown Atlanta, Georgia in Lake Meer from Piedmont Park.
Above: The Hilton Atlanta.

The CRU Sulphur 2017 conference returned to North America this year, moving to a new location in downtown Atlanta, Georgia. Attendance at what is now the 33rd sulphur conference was slightly down on the record highs of the past two years, at 430 delegates, but still substantial compared to Sulphur conferences of the past, and hopefully a sign of the industry's continuing health.

Market papers

The initial market session was led off as has become traditional with a look at oil and gas markets, this time presented by James Preciado of the US Energy Information Administration (EIA). Global oil markets are relatively balanced as 2017 moves into 2018, he said, with Brent Crude prices looking to be at around $54/bbl in 2018. The final quarter of 2017 would be the tightest point for markets.

Non-OECD Asia continues to drive liquid fuel growth, especially China and India, with some additional demand in Russia, Brazil and the US. OECD Europe, conversely, continues its pattern of long-term decline. New oil production should add around 1.7 million bbl/d of capacity in North America, mostly tight oil, but longer term most additional volumes will be available from OPEC producers, especially Saudi Arabia, Iran and Iraq. The US is still a net exporter (albeit at small volumes) under most long term predictions.

The spread between Brent Crude and Maya/Dubai prices fell in 2017, due to higher availability of light, sweet crude from Libya and Nigeria and less heavy, sour crude from Mexico. In the short term this affects the margins of more complex refineries as compared to less complex. However, current OPEC quota discipline is only about 85%, and as the oil prices rises more heavy oil is likely to be drawn back onto the market. There is also the effect of the 2020 IMO bunker fuel regulation to consider, as refiners struggle to meet extra demand for low sulphur fuel oil – simpler refineries will need more sweet crude, and this is sure to widen the sweet-sour price differential once more.

Considerable uncertainty remains over future forecasts; OPEC outages are currently at a 4-year low, and if they were to revert to the mean this would withdraw considerable volumes: OPEC surplus capacity is mainly in Saudi Arabia, and is both low (<1.5 million bbl/d) and declining.

Sulphur markets

Peter Harrison of CRU gave a summary of sulphur markets. Sulphur markets saw a price spike in Q3-4 2017, much of which seemed to be down to Chinese demand. Chinese imports of sulphur were down by 16% in the first half of 2017, but rose by 11% during 3Q 2017, and looked to be set to reach 11.2 million tonnes for the full year. The price rally could not continue, he said, and a decline was sure to come during 2018, but the precise timing could depend on the timing of new supply, e.g. from Kashagan or Qatar.

Elsewhere, the total global traded volume of sulphur was only up 600,000 t/a in 2017 over 2016, with Morocco, Brazil and Indonesia all seeing more imports to make phosphate fertilizers, but India and China seeing a decline overall (the latter due to increased domestic supply) and Australia requiring less sulphur due to the closure of nickel leaching operations. On the export side, Canada continued its long-term decline, and while production was stable in Russia new local demand meant exports fell. Japanese and Korean refineries were running at lower rates, leading to less molten sulphur availability, while Kashagan in Kazakhstan was now not due to be producing sulphur until 2018.

Overall, sulphur demand grew faster than supply during 2017, but overall production growth is past its peak. For the medium term, he forecast that demand would rise from 63.7 million t/a to 70.8 million t/a, with Morocco, Saudi Arabia, India and Russia all requiring more to make phosphoric acid. In China, some substitution of pyrite-based acid by sulphur burning acid is expected. On the production side, Alberta sour gas sulphur production continues to decline, but new refining and oil sands production should roughly match this. Russia is also seeing a similar growth in refining offsetting a fall in gas-based supply, with the outlier of the Norilsk nickel to sulphur project in Siberia.Central Asia is seeing new production from Tengiz and Kashagan, as well as Turkmenistan and Uzbekistan, but the Middle East will see the largest production increases, with the Wasit and Jazan projects in Saudi Arabia and potentially Fadhili in 2021, Kurait's Clean Fuels Project, Barzan in Qatar, and an expansion at Shah in the UAE. In Iran, the final phase of South Pars is due in 2022. Chinese sour gas production has had difficulties and timelines have been extended. Refinery growth is also slowing, with a limited project pipeline after 2021. Overall the market would see a surplus over the coming five years of around 300-900,000 t/a for each year, but stock-building could easily take care of this (especially at Norilsk) and project delays on both the production and consumption sides could change this figure considerably.

Sulphuric acid markets

Brendan Daly of CRU looked at sulphuric acid markets. Prices had rebounded in 2017, he said, with supply disruption in Q1 due to floods in Peru, smelter maintenance in North America and Chile in Q2-3, and smelter issue in Brazil during the first half of 2017 as well as later in Japan and Korea. This had led to more sulphur burning to make acid, while a rebound in the copper market had led to increased demand for acid in Chile. Morocco is continuing to import record volumes of acid, and Brazilian imports have also climbed as local supply has underperformed. Morocco will see some substitution with local sulphur burning capacity, while there will be less availability in North America, with increased demand for offshore acid in spite of a decline in demand for copper leaching.

On the phosphate side, Alexander Derricot of CRU present the market outlook paper. Demand is increasing in India and Brazil with better productivity due to increased irrigation in the former and an increase in cultivated area in the latter, but Chinese demand is falling due to a move away from corn and changes to government subsidies. There is also increased demand in Africa and Indonesia. On the production side, new capacity in Morocco, Tunisia and Saudi Arabia (amongst others) is likely to be balanced to a large extent by forecast closures by Agrium and Mosaic (Redwater and Plant City) as well as firm and potential closures in China. There will also be new capacity in Brazil and Turkey, as well as the potential for speculative, disruptive projects in Africa and China. Egypt is also moving to increase downstream phosphate production. China is in a very difficult period, however, Alexander said, with a falling domestic market and increased environmental regulatory pressures, as well as costs rising for both ammonia to make DAP and in terms of coal and labour. Companies have been forced to move operations away from the Yangtse River, in Hubei by 2020 which will impact 2.6 million t/a of NPK capacity. The largest eight producers have so far cut production in concert to preserve prices, but whether they can maintain this market discipline is open to question. Prices are likely to be low in 2018-19 due to new capacity, but China continues to play the role of marginal producer, and capacity closures should see prices rise towards 2022.

The North American sulphuric acid market was described by Kunal Sinha, CEO of Glencore's NorFalco subsidiary. The North American market totals 42 million t/a of sulphuric acid consumption, he said, of which around 30 million t/a is produced for local, captive use, mainly for fertilizer production, as well as some mining. The remaining 12 million t/a is 'merchant' acid, produced either by smelters (around 9 million t/a) or from regeneration of spent acid from refineries and used mainly in refineries (3 million t/a) or water treatment or other industrial uses, including food, pharmaceuticals, batteries, pulp and paper and speciality chemicals. Supply of acid from Vale in Sudbury, Ontario is falling due to a scrapping of the copper circuit and an overhaul of the nickel circuit there to meet new environmental regulations, reducing production by around 25% or 150,000 t/a. Demand is conversely increasing as copper leaching operations start up, and new fertilizer demand is expect in the Mid-West. This is likely to tighten the North American acid market by around 300,000 t/a overall (increased demand and reduced supply).

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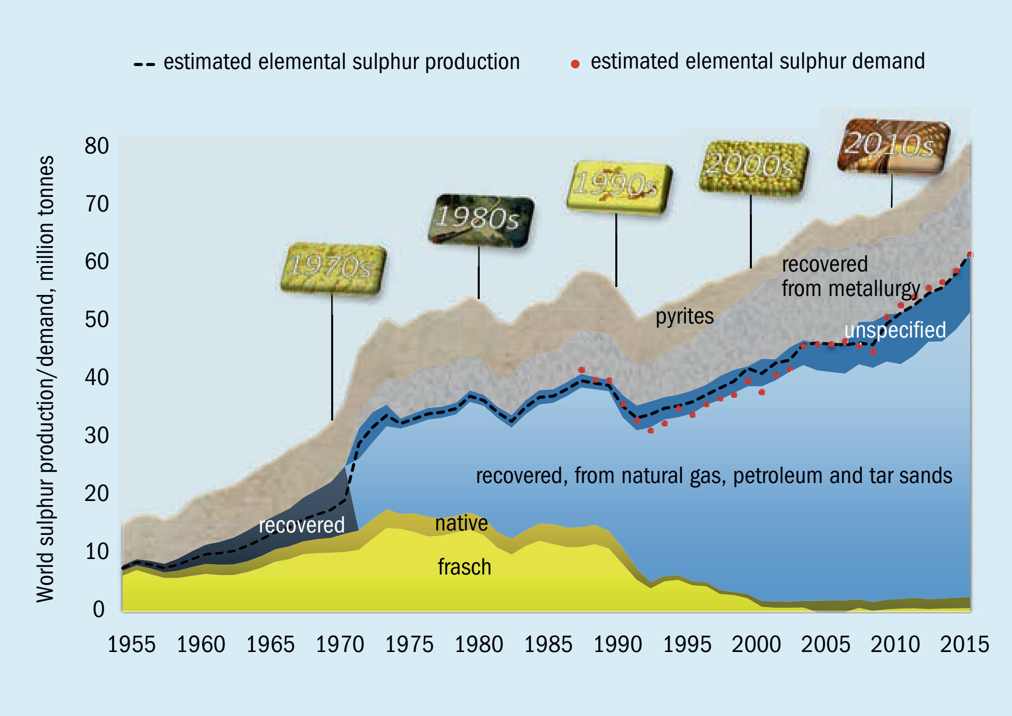
IMO sulphur cap

Two papers on Tuesday afternoon examined in some detail the impact of the International Maritime Organisation's forthcoming cap on the sulphur content of bunker fuels in 2020, by James Corbett, professor of Marine Policy at the University of Delaware and member of the IMO steering committee, and Olivier Kenter, a global manager at Shell Strategy. This topic is covered in much greater detail in the article on pages 20-22 of this issue.

Sulphur – This Is Your Life

Tuesday afternoon saw a light-hearted look at the history of sulphur, the sulphur and sulphuric acid industries, and the developments in them, chaired by Angie Slavens, and including almost 1,600 collective years of sulphur expertise seated at the table, including Rob Marriott and Paul Davis of ASRL, Gene Goar, Elmo Nasato, Randy Hauer and many more. Beginning with the formation of sulphur in the heart of early stars and moving through its role in Earth's geology and biology, the session finally took the sulphur industry in decade by decade slices from the 1960s to the present (see Figure 1), highlighting the developments which have shaped the industry as we know it today. It is hoped that the presentation from this session can ultimately become a resource for young engineers in the sulphur industry.

Fig. 1: **The changing sulphur industry, from Sulphur – This Is Your Life**



Technical papers – sulphur

The technical sessions began on Wednesday. In the sulphur strand, Claus plant papers perhaps unsurprisingly dominated, beginning with Gordon Finnie of Finnie Engineering describing how he had improved a company's SRU availability across 19 sites from 84% to 96% through a thorough benchmarking, root cause analysis, untangling of different standards for HAZOP and Level of Protection Analysis, introducing key performance indicators and setting safe operating and safe design limits. Christopher Filoon of Zeeco discussed flame detection in SRUs; the different types of sensor and their relative merits and shortcomings.

Aspen Technology and Sulphur Experts highlighted three common issues in SRUs which can be solved via simulation, including optimisation of the first bed Claus reaction versus the hydrolysis reaction; optimising the dewpoint margin; and avoiding catalyst deactivation. Simon Weiland of Optimised Gas Treating similarly looked at how ProTreat analysis software can help optimise Claus waste heat boiler (WHB) economics, important given the many and often competing factors going into the performance and reliability of the heat exchanger.

Domenica Misale-Lyttle of Industrial Ceramics showed via the usual salutary case studies how WHB operating parameters need to be carefully managed to ensure correct heat transfer in the WHB tunes. Failure to do so can lead to high temperature H_2S corrosion of the tubesheet protection ferrule system.

Rob Marriott of ASRL presented on ASRL's continuing investigations into ammonia destruction in the Claus furnace, with the aim at improving modelling for new and existing furnace/burner design. While the experiments confirmed that a duel zone furnace design worked better for ammonia destruction than a single pass, this was believed to be a mixing effect, and Rob theorised that it would be possible to design burners to achieve dual mixing zones in a straight through furnace design.

Other papers looked at how to avoid hydrocarbon carryover into sulphur plants; design and control of a two stage sour water stripper; and mitigation of elemental sulphur deposition in sour gas petroleum reservoirs. Crescent Technology also described their design work on the Mosaic New Wales sulphur melter, and Sauereisen Inc also presented a duel lining system for the rehabilitation of molten sulphur storage. The final paper of the sulphur technical session addressed the design of API 610 pump design, a fuller discussion of which can be found on pages 42-46 of this issue.

Tail gas treatment

A number of papers looked at the issue of SRU tail gas treatment. Marco van Son of Shell began by introducing Shell's new SCOT (Shell Claus Off-gas Treating) ULTRA system, which offers a step change in performance from previous SCOT systems due to the inclusion of the *JEFFTREAT ULTRA* solvent family, developed jointly by Shell and Huntsman. The technology also features Criterion Catalysts' C-834 catalyst, which provides high activity at low temperature and which improves the conversion of organic sulphur compounds like COS.

Another improved solvent was the topic for German Oliveros Patino of Dow Chemicals. This one, *UCARSOL*, has been developed jointly between Dow and the China National Offshore Oilfield Company (CNOOC) and trialled in a TGTU operated in Guangdong. Considerable improvement over straight MDEA was demonstrated, and the TGTU was comfortably able to meet the new Chinese emissions limit of 100 mg SO_2/Nm^3.

Sulphur Experts' Joe Brindle described what he called the 'seven deadly sins' of tail gas treatment units and how to avoid them to achieve high recovery rates – poor reaction stoichiometry; catalyst deactivation; operating the first converter too cold; operating the second and third converters too hot' by-passing gases around conversion stages; a high final condenser temperature; and liquid sulphur entrainment.

Bob van der Giessen of EuroSupport emphasised the benefits of using titania instead of alumina in Claus and tail gas catalysts, including improved low temperature performance, resistance to gas contaminants and lifetime, making it suitable for high temperature hydrogenation applications that suffer from insufficient energy.

The final paper of the session was presented by Mahin Rameshni and offered a variety of ways of reducing SO_2 emissions from sulphur plants.

Technical papers – sulphuric acid

The sulphuric acid technical session began with updates on new acid plant designs. Stefan Brauner of Outotec examined the impact of what the German government calls 'Industrie 4.0' on acid plants – the current trend of automation, data exchange, cloud computing, cognitive systems, and linked cybernetic systems or the 'internet of things', with reference to some case studies of how these are impacting on current plant design trends, such as the PORS Plant Operability, Reliability and Safety analysis tool as part of intelligent analytics to assist plant operators, which may ultimately form part of a creeping automation of chemical plant operation. He also addressed some of the challenges facing acid plant designers, such as falling ore grades in the metal and mining industry, environmental concerns over cadmium, mercury and other heavy metals in the phosphate industry, and tightening environmental regulations on SO_2 emissions.

Rene Dijkstra of Chemetics presented their *CORE* (Cooled Oxidation REactor) approach to acid plant design – a rebranding of the *BAYQIK* technology Chemetics acquired from Bayer in 2016. CORE continuously removes reaction energy from the reactor, maintaining the catalyst temperature in the optimum range. And in the new *CORE-S* design, air cooling is replaced with molten salt, allowing for lower temperatures in the reactor, and improving equilibrium. This also leads to lower power consumption and a smaller footprint, and can generate higher SO_2 concentrations at lower cost. Air cooling via the basic *CORE* system is still preferred for smaller capacity plants (<600 t/d) he said, but *CORE-S* provided for larger designs of 2,000 t/d and up.

MECS view of the future of sulphuric acid technology was presented by Garrett Palmquist. The *MAX3*™ technology combines two proven concepts – MECS' *SolvR* regenerative solvent which has high SO_2 capacity at high temperature and low capacity at low temperature, allowing it to absorb SO_2 in one part of the process and release it elsewhere, and which can facilitate energy recovery and lower SO_2 emissions, and the *SteaMax* steam injection system. Maximising steam injection into the heat recovery system upgrades the steam to high pressure (60 bar), leading to higher power production.

> **"The continuing focus on SO_2 as a pollutant in all walks of life shows no sign of abating.**

On the catalyst side, BASF described new developments in their sulphuric acid catalyst line, and Haldor Topsoe presented their LEAP5 SO_2 oxidation catalyst, both of which are detailed in our sulphuric acid catalyst article on pages 47-52.

Sulphur dioxide emissions

The continuing focus on SO_2 as a pollutant in all walks of life shows no sign of abating, and sulphuric acid plants are no exception. Consequently several papers looked at reducing and managing SO_2 from an acid plant. Paolo Olis of Mosaic and Nicolas Edkins of Shell Cansolv compared real world data from two of Mosaic;s acid plants in Louisiana, one a single absorption plant equipped with a Cansolv system, the other a conventional double absorption unit. The Cansolv system has proved to be not only competitive in terms of SO_2 emissions but also on a project life cycle cost basis compared to alternative solutions.

Brian Lamb of MECS looked at SO_2 emissions in Europe, the US and China, and their health and environmental costs, as well as remission strategies and their relative costs and benefits.

NORAM Engineering's take on emission reduction strategies focuses primarily on tandem or parallel sulphuric acid plants. As the plants rarely start up at the same time, the technology transfers emissions from a plant undergoing unsteady state operation to a neighbouring plant at steady state, avoiding the emissions peak at start-up that is an issue for acid plants.

What do blast furnace coke and modern sulphuric acid plants have in common, asked Zion Gupta of thyssenkrupp Industrial Solutions? It isn't a joke with a punchline – tkIS manufacture both, and have sought to apply solutions from the coke industry to the sulphuric acid industry, including safe configuration of the heat recovery system, improved plant layout, and a closed loop start-up system.

Other acid papers

The caustic nature of acid plants requires extremely resistant materials. Magneco/Metrel have developed and patented the use of colloidal silica as a binding matrix for monolithic refractories, now sold as the *Metpump* brand of linings, inherently resistant to acidic conditions and greatly reducing attack and degradation by thermal shock, elevated temperature and mechanical loading.

While SO_3 process gas dewpoint is usually well below process gas temperatures, there is the potential for moisture leakage from the drying tower, through the final stage of conversion and economiser outlet, leading to conversion to acid and downstream corrosion. Early detection of such leaks is therefore crucial. Breen Energy Solutions has collaborated with the acid industry to measure process gas dewpoint at the converting tower economiser outlet.

Another measurement paper, by SensoTech, covered monitoring sulphuric acid and oleum strength with a single measuring device, which incorporates *LiquiSonic* sonic velocity analysers and a second measurement technology such as density measurement to gauge the oleum strength.

Lastly, Shixue Chen of Wylton Dazhou Chemical Co in Sichuan, China described online pressure welding repairs to a leaking converter.

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STERCORAT
Production of Stercosul® – ATS liquid fertiliser

STERCORAT Hungary Kft is pleased to announce the building of a new site for the production of Stercosul® liquid fertiliser in Slovakia. Production will start at the end of the first quarter 2018.

✔ **STERCORAT with strong 'know-how'** will utilise a unique ThioSolv® SWAATS technology at SLOVNAFT Refinery.

✔ **Stercosul® - ATS liquid fertiliser for the maximisation of crop return and its high quality**
Stercosul® enables you to achieve the full potential and higher quality of your crops by adding sulfur through this liquid fertiliser.

PRODUCT:
- 26 % sulfur, 12 % nitrogen, 60 % aqueous solution
- Non-flammable

PACKAGING:
- Freely loaded into cars and rail tanks

✔ **Flexible and reliable partner**
Our production capacity of up to 50 000 MT per year and a large storage capacity of up to 10 000 m³ make us a highly flexible and reliable partner.

If you are interested, please contact us, as we will be glad to meet with you and discuss in detail the possibility of any cooperation.

You can contact us on info@stercorat.eu

More info www.stercorat.eu

Keeping the conversation flowing

The 4th Annual Middle East Sulphur Plant Operations Network Forum (MESPON 2017), organised by UniverSUL Consulting and supported by ADNOC took place in Abu Dhabi, UAE, 15-17 October 2017.

Each year the attendance at MESPON has been steadily growing, with approximately 250 delegates comprising more than 60% operators the event is fast becoming one of the industry's premier events for technical knowledge sharing of sour gas treating and sulphur recovery operating experience. The aim of the MESPON forum is to 'connect the dots' – to maximise utilisation of current experience and expertise in the Middle East via networking and to provide access to the knowledge and resources of the global sulphur community across the globe from Western Canada, to Europe and the Far East.

Welcome remarks were given by MESPON's new executive chairman, Omar Al Marzooqi of ADNOC Sour Gas (formerly known as Al Hosn Gas).

This year's three day agenda featured over 15 technical presentation and multiple panel sessions focusing on the design and operational challenges and considerations for amine plants, sulphur recovery units, tail gas treating unit and sulphur handling facilities in the Middle East. A key element of the programme, the annual MESPON roundtable, which addresses relevant current and regional operations issues, took place on the final day of the event.

The forum agenda was split into six sessions:

- **Session A:** Where we've been and where we're going…
- **Session B:** Oxygen enrichment – emergence in middle eastern gas plants
- **Session C:** Practical considerations for waste heat recovery from SRUs
- **Session D:** Plant optimisation for performance and cost savings
- **Session E:** Continuous improvements in sulphur handling
- **Session F:** Developments in tail gas treating

The Middle East is the largest sulphur producing region in the world, producing 17.3 million tonnes of sulphur (28% of world production) in 2016. Throughout the region there is strong focus on operational excellence, i.e. asset optimisation, continuous improvement, technology advances, and lean organisations but with no HSE compromises. Many of these elements were discussed at the forum.

Asset optimisation

Maximum throughput study

Muna Al Maazmi and Mahmood Al Murid of ADNOC Sour Gas shared the results of a maximum throughput study for their sulphur recovery and sulphur granulation plants. ADNOC Sour Gas first looked at increasing throughput of its sulphur recovery units to 110% using the design margin, i.e. with no investment required, and then evaluated increasing the SRU throughput to 120% with minimum investment. The sulphur production facilities consist of 4 x 25% identical parallel sulphur recovery units with each unit designed to treat 115,900 Nm³/h of acid gas to produce 2,500 t/d of liquid sulphur.

Other SRU design features include:

- Feed gas is from solvent regeneration units (DGA) and Selexol units
- Fluor licensed two-stage Claus plant with hydrogenation/amine tail gas treating
  - BTX destruction
  - Dual thermal stages in each train
- ExxonMobil's FLEXSORB® SE Plus in the TGTU section
- D'GAASS liquid sulphur degassing technology
- Sulphur recovery: 99.9%+
- SO_2 in stack gas: < 500 mg/m³

The plants are already operating at well above design capacity making them the world's largest sulphur plants. In the study, test runs showed no limitation in any SRU at 110% capacity (2,730 t/d).

Evaluation results for 120% SRU with minimum investment showed maximum acid gas feed per train can be achieved at 133,850 Nm³/h with total liquid sulphur production of 2,977 t/d. The main process equipment, control valves, relief valves and piping are adequate for increased plant capacity. The licensor has endorsed the company's intention to operate the units at this capacity. The following main modifications and operational changes will need to be implemented:

- sulphur seal replacement;
- third condenser PSV replacement with higher discharge capacity;
- blowers operation at rated capacity;
- minimum fuel gas co-firing only for burner protection.

The study also included an evaluation of the sulphur granulation plant which consists of ten 50 t/h Enersul GX granulators. The test evaluation results showed that the granulation rate can be increased to 60 t/h without affecting the unit integrity, maintaining reliability and availability of the unit and without adding new GX units.

Continuous improvements

Improved SRU shutdown procedure

Hamad Al Ali of ADNOC LNG (formerly known as ADGAS) and Jamie Swallow of Sulphur Experts discussed improvements that have been made to the shutdown procedure at ADNOC LNG Das Island sulphur recovery units (SRUs). The SRUs at ADNOC LNG comprise three trains (all 3 stage modified Claus units). Trains 1 and 2 are both mid 1970s units with a capacity of 550 t/d each and are equipped with fired reheaters. Train 3 is a 1993 unit with a capacity of 500 t/d, equipped with steam reheaters, and with titanium dioxide in bed 1 of the converter. A dedicated SUPERCLAUS® stage was added to all units in 2006. The main SRU shutdown challenges are:

- non routine operation – turnarounds are now typically every five years which means operators often have limited experience of shutdowns;
- extensive procedure – all sulphur needs to be removed to avoid plugging;
- high risk operation – higher than normal gas temperature and the risk of temperature excursions and fires.

In the old ADNOC LNG shutdown procedure steps were carried out according to the original manual/licensor:

- catalyst preparation (heat soak) with acid gas firing;
- sulphur removal with fuel gas firing;
- regeneration with fuel gas firing, the reheater was kept on while introducing a "controlled" amount of oxygen when the catalyst beds were hot;
- forced cool down.

The old regeneration procedure was time consuming lasting approximately 72 hours and increased the risk of equipment damage and unit trips.

By contrast, the new ADNOC LNG procedure reduces the shutdown time to 48-72 hours. The regeneration step has been removed and cool down takes place in two steps:

- Reheater switched off on completion of sweep:
  - main burner sub-stoichiometry is maintained;
  - bed cools down to approximately 150-160°C.
- Cool down:
  - air to fuel ratio or excess oxygen value of main burner is slowly increased;
  - condensers are drained once temperature drops;
  - nitrogen is added to enhance further cooling.

When the bottom rows of condenser tubes get plugged with sulphur they are difficult to clean, extending the shutdown time and requiring hydro drilling. In the new shutdown procedure, ADNOC LNG have introduced a new "back steaming" procedure at the end of the cool down operation:

- air flows through the unit;
- water side of condensers are drained to prevent reheating of process gas;
- live steam is added to the condenser water side to increase the condenser temperature (165°C) which remelts sulphur from the bottom rows.

As a result, the bottom rows are absolutely clean. Back steaming adds approximately half a day to the shutdown procedure but reduces the turnaround duration by 5-7 days (no need for hydro drilling/cleaning).

Further improvement of the shutdown procedure is possible and ADNOC LNG is looking at a number of areas:

- Ensure substoichiometric firing at shutdown:
  - air to fuel ratio check at start-up;
  - use of oxygen analyser;
- Increase fuel gas firing to speed up the procedure:
  - currently at 10-12% of the design plant flow;
  - optimum is 20-30% of the design plant flow.
- Nitrogen availability to speed up final cooling of reactors:
  - warm ambient air does not cool;
  - looking at using more nitrogen to speed this up.

Technology advances

Novel TGTU technology

Adel Seif El Nasr of ADNOC Gas Processing (formerly known as GASCO) introduced to delegates a novel temperature swing absorption (TSA) Claus tail gas treating technology currently under early stages of research and development. It is an absorption based process (using a mixed oxide adsorbent) in which SO_2 is regenerated and sent back to the Claus unit. The starting point is a plant already achieving 99% sulphur recovery, e.g. with the CBA process. The R&D project has just completed the research stage and is entering the technology development stage, which is expected to last for 2-3 years, before entering the field deployment stage.

The main project motivation and drivers can be summarised as follows:

- Utilisation of Best Available Technologies (BAT):
  - to reduce SO_2 from the asset's vented gas (target 150 mg/Nm³);
  - can be applied in existing assets, e.g. Habshan 1 & 2.
- Fostering innovation across ADNOC through the development of own technologies:
  - to grow ADNOC and for maximisation of value;
  - to develop a competitive edge over existing best in class technologies e.g. amine-based TGTU technology currently deployed in Habshan 5.
- Tackling the upcoming challenges of tomorrow:
  - no mandate exists in the UAE to achieve very low levels of SO_2 emissions;
  - UAE is a signatory on the COP mandate which will create more ambitious future targets;
  - anticipation of government mandate to reduce SO_2 emissions from existing assets.

Performance of the adsorbent after pelletisation and during regeneration is to be proven at the next stage of development. ■

Oxygen enrichment for grassroot SRUs

The almost unanimous reason cited for not deploying oxygen enrichment for grassroot sulphur recovery units (SRUs), despite the manifest benefits, is that "no oxygen was available at the site." It is this misconception leading to a missed opportunity for tremendous value creation that **Uday Parekh** of Unpaar Performance LLC addresses in this article.

Oxygen enrichment is a well-established technology for increasing the capacity of SRUs and has been practiced commercially for over 30 years with more than 150 SRUs having deployed this technology worldwide. However, virtually all of these projects have been for the retrofitting of existing SRUs with very few grassroots oxygen-enriched SRUs, except for the niche sector of coal gasification wherein incremental oxygen is available very economically.

Every new refinery project or significant refinery expansion requires large quantities of nitrogen. By building a co-product air separation unit (ASU) that will produce both nitrogen and oxygen, the cost of the oxygen required for SRU oxygen enrichment becomes extremely low. This cost is offset many times over by the capital cost savings from building fewer or smaller SRUs. Operational savings through reduced power consumption, preheating/co-firing and incinerator fuel gas consumption further improve the economics of oxygen versus air-based SRUs.

This article provides comparisons of the costs of grassroots oxygen versus air-based SRUs. SRU redundancy as required for most projects is achieved much more economically via oxygen enrichment versus having to build larger or spare SRUs wherein the costly spare capacity is only utilised during outages of sister units. The cost of O_2/N_2 supply is based on inputs from some of the leading industrial gas companies worldwide. All major gas supply options (merchant, on-site, pipeline, adsorption and cryogenic) are covered so that project owners and licensors can make the most informed decision on SRU technology. With the benefits of oxygen enrichment well accepted in the industry, this article demonstrates that a prior presence of oxygen supply is not necessary for adopting grassroots oxygen-based SRUs.

PHOTO: AIR PRODUCTS

SRU oxygen enrichment background

Oxygen enrichment of the combustion air to the SRU reaction furnace is a proven, economic, reliable and safe method for addressing the dual needs of increasing SRU capacity while simultaneously conserving capital for more profitable operations. The typical SRU reaches its ultimate sulphur production capacity when the maximum allowable front-end pressure prevents further increase in feed rate. Oxygen enrichment reduces the flow of process gases by reducing the quantity of nitrogen that enters with the combustion air. This reduction in process flow rate allows a corresponding increase in SRU acid gas feed rate and subsequent increase in sulphur production.

In the Claus process about one-third of the hydrogen sulphide in the acid gas stream is combusted to sulphur dioxide which further reacts with the remaining hydrogen sulphide to form elemental sulphur and water in the vapor phase. The combustion reaction and approximately 60-70% of the conversion of hydrogen sulphide to sulphur take place in the thermal reactor with the remaining conversion of hydrogen sulphide to sulphur taking place in a series of catalytic reactors. Representative reactions are summarised below:

Combustion reaction:

$$H_2S + {}^3\!/_2\, O_2 \rightarrow SO_2 + H_2O$$

Claus reaction:

$$2H_2S + SO_2 \rightleftharpoons 3S + 2H_2O$$

Overall reaction:

$$3H_2S + {}^3\!/_2\, O_2 \rightleftharpoons 3S + 3H_2O$$

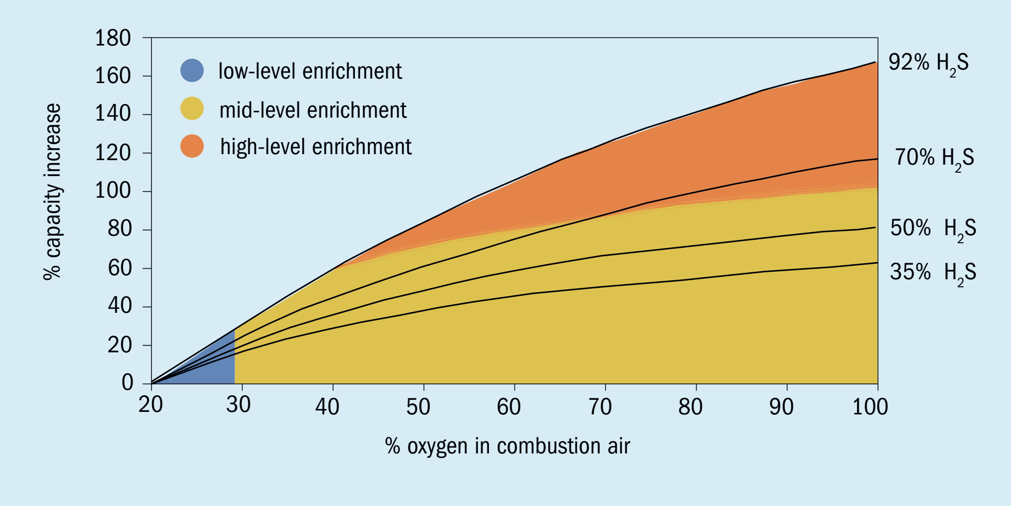
Stoichiometrically, 100 kmol/h of hydrogen sulphide requires 50 kmol/h of oxygen. If all of the oxygen is provided by the air, 189 kmol/h of nitrogen accompanies the 50 kmol/h of oxygen. This nitrogen (over 50% by volume in the feed) contributes to a large amount of the pressure drop through the SRU. Oxygen-enriched operation reduces the amount of nitrogen entering the process. Table 1 shows how nitrogen is replaced by acid gas while keeping the total molar/volumetric flow rate (and hence the pressure drop) through the SRU equal or less than in the air-based case at varying levels of oxygen enrichment. Acid gas throughput to the SRU is dramatically increased. This is the underlying principle for the effectiveness of oxygen enrichment

Table 1: **Why oxygen enrichment increases SRU capacity**

Oxygen enrichment, %	20.9 (air)	25	50	100
Acid gas, kmol/h	100	113	170	226
Oxygen, kmol/h	50	57	84.5	113
N_2+Ar, kmol/h	189	169	84.5	0
Total flow to reaction furnace, kmol/h	**339**	**339**	**339**	**339**
Total flow to TGCU, kmol/h	293	286	261	235

Total flow constant; Acid gas flow increases as O_2% increases

Fig. 1: **Oxygen enrichment technologies**



as a debottlenecking solution in the refining, chemical and other process industries.

Oxygen enrichment technologies

There are three distinct SRU oxygen enrichment technologies depending on the capacity increase desired. These are depicted in Fig. 1 and summarised below

Low-level oxygen enrichment (LLE)

In low-level oxygen enrichment (LLE), oxygen is injected into the combustion air process line at an appropriate safe location through a custom-designed diffuser which provides good mixing and oxygen safety. Considerations related to oxygen compatibility and cleanliness of the air main and other components usually limit this LLE technology to enrichment levels of about 28%. This technology is relatively easy to implement but capacity increase is limited to about 25% or a little higher for rich acid gas streams.

Mid-level oxygen enrichment (MLE)

Higher levels of oxygen enrichment beyond 28% require a dedicated pathway for the oxygen due to oxygen safety, flame stability and other considerations. This technology, termed mid-level oxygen enrichment (MLE), requires the use of a special burner with discrete oxygen port(s) to safely handle oxygen. The air and oxygen are not premixed as in the LLE technology because of material compatibility concerns and enter separately via a specially designed burner. The upper limit of this technology is set by the temperature limitations of the furnace refractory (about 1,550°C) and this technology can provide capacity increase of up to about 50-60% at oxygen enrichment levels of 40-45% for typical refinery acid gas streams – subject to some equipment debottlenecking if necessary.

High-level oxygen enrichment (HLE)

Even further capacity increases (doubling or more of SRU capacity) can be provided through deployment of temperature moderation technologies. These technologies termed high level oxygen enrichment (HLE) achieve temperature moderation via a) recycling cooler gas from downstream into the reaction furnace – the COPE® process developed by Air Products and GAA and now offered by Fluor, or b) staged combustion – the SURE™ process offered by WorleyParsons and Linde or c) the use of a proprietary burner to control maximum furnace temperature – the OxyClaus™ process offered by Air Liquide.

One important point to note is that the MLE to HLE boundary marked by the need for temperature moderation is applicable mainly to the rich acid gas streams found in refineries. SRUs in gas plants and gasification complexes with typically low H_2S concentration can safely use 100% oxygen without hitting the SRU reaction furnace temperature limit and therefore do not need temperature moderation and may in some instances still require fuel gas co-firing to achieve adequate temperatures for BTX destruction. This is seen in Fig. 1 where the lines for 35% and 50% H_2S are entirely within the regime designated as MLE even at 100% oxygen.

General benefits of oxygen enrichment

These have been very well covered in the industry literature and are therefore only listed briefly here for reference:

- capacity increase;
- full redundancy in case of a planned or unplanned shutdown of a sister SRU;
- capital cost savings;
- operational flexibility to handle higher upstream througputs or more sour crudes/feed gas;
- operational reliability via better contaminant destruction (NH_3, BTX), better flame scanner operation, etc;
- improved conversion and reduced emissions;
- hotter flame and better contaminant destruction;
- quick implementation;
- compact footprint;
- proven safety.

Main equipment impact

These have also been well covered in the industry literature, including in case studies, so only a very brief summary is provided below of equipment that is or may be impacted:

Burner: A burner with a dedicated oxygen pathway is required due to material compatibility considerations. In certain designs the air burner can be used with a changeout of the center gun to one with an oxygen pathway.

Waste heat boiler performance: In many instances the WHB is found adequate for capacity increases well into MLE. This is due to much better heat transfer including from the substitution of a non-radiating molecule (nitrogen) by a radiating molecule (water vapour, a product of combustion and the Claus reaction).

Effects on TGCU and incinerator: In the quench section, the condensing load on the quench tower and cooler increases more or less in direct proportion to the increase in sulphur throughput and this section generally needs to be debottlenecked if the increase in SRU capacity is more than a modest amount. The amine absorber has a lower feed gas flow and a higher partial pressure of H_2S, resulting in a lower quantity of H_2S in the absorber vent gas. Thus, there is less incineration fuel consumed, reducing operating cost as well as CO_2 emissions.

Oxygen supply options

Oxygen to a refinery or gas processing plant can be either a) delivered as liquid oxygen (LOX) by on-road tanker trucks or via pipeline as gaseous oxygen (GOX) or b) generated on-site on the plant premises or "across the fence" using non-cryogenic (adsorption) or cryogenic air separation technology. From a commercial standpoint, the latter mode of supply can be sale of equipment (SOE), wherein the operating company purchases and operates the oxygen plant, or can be sale of gas (SOG), wherein the oxygen supplier builds, owns and operates the plant (BOO). In the SOG framework, the operating company and the oxygen supplier would enter into a long term supply contract with a typical term of 10 to 20 years. The correct choice of oxygen supply is critical in obtaining the best economics and is a function of several different parameters.

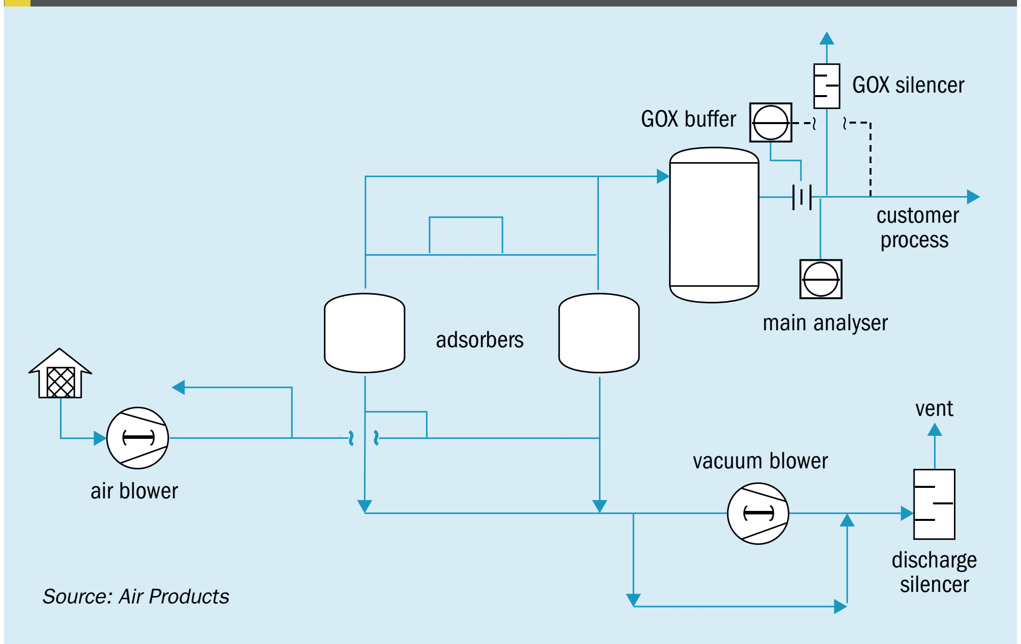
Liquid oxygen (LOX)

This mode of supply is the most flexible. Oxygen is delivered as liquid by truck, from one or more central manufacturing facilities. A cryogenic LOX storage tank is installed at the site, along with a vaporiser, by the oxygen supplier. Oxygen is withdrawn from the tank and vaporised as required to meet process requirements. The tank is sized to ensure that there is always sufficient capacity to meet demand and is refilled by tanker, as required according to tank volume information, communicated by telemetry to the LOX supplier.

Oxygen vacuum swing adsorber (VSA)

This mode of supply provides oxygen by on-site generation for oxygen requirements between those economically served by LOX and an on-site cryogenic plant on the lower and upper end respectively. The process uses a high-efficiency molecular sieve to selectively separate out oxygen from the feed air by adsorption. Oxygen VSAs operate in a batch process. During a cycle, the two adsorber vessels (Fig. 2) are alternately pressurised with air to produce oxygen, then evacuated under vacuum to remove nitrogen, moisture and carbon dioxide and regenerate the adsorbent. Oxygen for customer use needed during the portion of the cycle when oxygen is not being produced is supplied from the product buffer tank, maintaining an uninterrupted, consistent oxygen flow. The oxygen typically has a concentration of around 90 to 95% and is compressed to the desired process requirement. To provide an uninterruptible supply, a backup LOX tank is typically included in the supply configuration.

Fig. 2: **PRISM® oxygen VSA process**



Source: Air Products

PRISM® VSA oxygen generators.

Oxygen cryogenic plant (ASU)

This mode of supply provides oxygen extremely economically by on-site generation for large volume requirements. Air is separated by cryogenic distillation into oxygen and nitrogen based on their difference in boiling points of -297°F (-183°C) and -320°F (-196°C) respectively. The basic steps for this process are compression of the feed air, followed by pre-cooling and purification using a molecular sieve to remove the water vapour, carbon dioxide, hydrocarbons and other impurities in the air. This is followed by heat exchange against the ASU products, refrigeration via Joules-Thomson expansion and separation of the components by distillation.

Oxygen purity from the ASU can be customised based on the customer's need. For oxygen enrichment applications, 95% oxygen purity is often the most economical. Opportunities for integration exist when co-products can be used, such as high purity nitrogen and clean dry air (CDA) or oxygen for other applications at the same or adjacent facilities. Such approaches improve economics further. On- site cryogenic plants typically have LOX backup to ensure 100% supply depending on the customer's needs. This provides much more reliability than that of a typical SRU. The LOX tank inventory is maintained through a small amount of liquid production from the plant.

Cryogenic air separation plant.

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Pipeline

Should the customer site be located near an oxygen pipeline, this may be the most attractive mode of supply. Oxygen is generated off-site, by cryogenic plants, and fed to the pipeline. Available volumes will usually be high and prices low and often the supply can be provided with slightly more flexible terms since the oxygen supplier's investment is not entirely dependent on one customer.

Choosing the right oxygen supply

Table 2 provides a rough decision-making rubric for the correct oxygen supply option based on few key parameters.

As discussed in the introduction the very vast majority of SRU oxygen enrichment projects over the last 30+ years have been for retrofits. The most common scenarios for implementation are:

- an increase in refinery or gas plant throughput causing an incremental need in additional SRU capacity;
- sourer crude or gas causing an incremental need in additional SRU capacity;
- redundancy – maintaining SRU complex capacity when a sister unit has a planned or unplanned outage;
- extending run times when a SRU is facing pressure drop limitations so that the SRU turnaround can be synchronised with the turnaround of upstream facilities and not having to ratchet down plant production;
- as a temporary solution pending a better definition of future SRU capacity needs from upstream throughput/ feedstock changes.

All of these scenarios involve varying oxygen flow rates, often intermittent use and no clear picture for steady long term demands. This makes these situations very amenable for LOX supply, where supply contracts involve relatively low commitments in terms of monthly charges and minimum volumes. From the oxygen supplier's perspective these attractive terms can be offered because LOX supply requires relatively little fixed investment dedicated to the customer and the product is sold to a large customer base from central manufacturing facilities providing a portfolio effect and low offtake risk. Overall, oxygen enrichment via LOX is very compelling given the low cost of entry and that the benefits of SRU oxygen enrichment are many fold.

It is when the customer's SRU processing needs grows much beyond existing air-based capacity and the oxygen requirement and costs of LOX supply become significant that a key decision has to be made whether to build a new SRU or go the route of an on-site oxygen plant. Choosing between the two options requires a detailed comparison of the capital and operating costs. An on-site oxygen plant will require more of a commitment from the customer since in this case the supplier is making a dedicated investment exclusively for the customer. The customer can elect to buy an ASU and operate it (SOE) or elect across the fence supply (SOG). The rest of this article focuses on the costs of on-site oxygen supply (SOG, SOE) since this is the area requiring maximum illumination given that most projects go the route of air-based plant for new build and large retrofits suffering from the impression that SRU oxygen enrichment is not an option if oxygen is not already available at the site. Customers and licensors rarely investigate the costs of investing in on-site oxygen supply.

In SOG, the capex and operating costs both variable and fixed (opex) incurred by the oxygen supplier for the ASU as well as return on capital are reimbursed by the customer on a monthly/periodic basis for the predetermined length of the contract. The oxygen supplier builds, owns and operates the oxygen plant for the contract term. The oxygen price is typically held constant save for any escalation for power and other ASU inputs over the life of the contract.

Oxygen economics for four scenarios are provided, spread over a wide range

Table 2: **Choosing the right oxygen supply mode**

Supply Features	LOX	VSA	Cryo	Pipeline
Flow range, t/d	1-100	50-250	150+	100+
Price range, $/t	60-120	35-70	25-55	25-50
Commitment	low	high	high	medium
Coproduct N_2	no	no	yes	maybe
Time to implement, months	1-2	10-12	12-14	6-8
Location limitations	yes	no	no	yes
Application: best fit				
Flow	low	medium	high	high
Use pattern	variable	steady	steady	variable

LOX = liquid oxygen; VSA = vacuum swing adsorber; Cryo = cryogenic air separation plant; Pipeline = gas piped in from remote air separation plant.

Table 3: **Economics of SRU oxygen enrichment**

		GOX 1.8 barg	GAN 1 barg	GAN 10 barg	GOX cost	GAN cost	GOX NPV	Savings vs SRU*	
Case	Process	t/d	t/d	t/d	$/t	$/t	$ (million)	$ (million)	%
1	VPSA	200	-	-	66		36	29	45
2	Cryogenic	350	-	-	54		52	44	46
3a	Cryogenic	350	360	-	32	25	31	65	68
3b	Cryogenic	350	-	360	32	36	31	65	68
4	Cryogenic	1,750	-	-	36		172	128	43

** Savings will actually be larger since oxygen plant opex has been included in NPV but SRU plant opex has not.*
Assumptions:
Project in Middle East; FX rate: €/$ = 1.14; utilisation: 8,600 h/a; 15 year O_2 supply contract; discount rate: 10%, 1 t/d O_2 supplants 1 t/d of new SRU capacity.
Analysis based on indicative oxygen and nitrogen costs from Linde (Dr Marcus Guzmann).

Table 4: **Air-based vs grassroots COPE comparison**

	4 x 400 t/d air-based SRUs + TGCUs	3 x 300 t/d air-based SRUs + TGCUs + ASU for O_2 (& N_2)	
Capital cost, $ million	base	base - 90*	
Yearly operating cost, $ million			
Power (8c/kwh)	base	base + 1.1	- $0.4 million
Natural gas ($0.35/Nm³)	base	base - 3.0	
Oxygen	none	+ 3.2	
Operations & maintenance	base	base - 1.7	
Emissions (t/a)			
SO_2	base	base - 59.7	
CO_2	base	base - 23,500	

** +/- 25% USGC basis; all numbers are indicative estimates* *Source: Air Products/GAA*

Table 5: **Air based versus O_2-based 5,000 t/d capacity SRU/TGTU**

	Air only 4 SRU trains	O_2 enrichment 2 SRU trains
Differential total installed cost[1]	base	base - 50%
Differential net capital cost[2]	base	base - 44%
Differential operating cost (NPV)[3,4]	base	base + 21%
NPV of total savings for 20 year life cycle[5]	base	base - 40%

Notes:
1. Does not include cost of an oxygen supply system.
2. Considers cost of new, dedicated oxygen supply system.
3. Operating cost based on steam cost of $6.17/tonne, power cost of $24.50/MWh, and 8,000 operating hours per year.
4. NPV based on a 20 year plant life, an 8% discount rate, and 2% per year escalation.
5. Considers capital cost and operating costs for a 20 year plant life cycle. *Source: Fluor*

of oxygen volume requirements so as to cover a) both on-site oxygen generation technologies – non-cryogenic (adsorption) and cryogenic, b) absence and presence of co-product nitrogen and c) oxygen volumes covering the range from the requirement for a relatively small size refinery/gas plant to volumes relevant for SRUs at the world's largest gas processing plants.

The four scenarios summarised in Table 3 are:

1. Oxygen production via a VPSA: 200 t/d which can be used to roughly double the capacity of one 200 t/d SRU or two 100 t/d SRUs – applicable to several relatively small/sweet refineries or gas plants.
2. Oxygen production via an ASU: 350 t/d which is on the smaller end of cryogenic technology and applicable to the capacity needs of small to medium size refineries and gas plants.
3. Same oxygen production as Scenario 2 but with co-product nitrogen to illustrate the sharp decrease in oxygen price (40+%) when credit is obtained for nitrogen use at the same or proximate facility.
4. Oxygen production at relatively high volumes: 1,750 t/d though this is just about one-third of the demonstrated high end of ASU capacity. It can be seen that the unit O_2 price is 55% of the VPSA and 67% of the 350 t/d ASU demonstrating the strong impact of economies of scale.

Analysis

The results from the analysis are striking in terms of the very strong economic superiority of O_2-based SRUs compared to air-based SRUs across a 9-fold spectrum in size ranging from 200 t/d to 1,750 t/d O_2 supply. The analysis is based on HLE and each tonne of O_2 on average providing a tonne of extra SRU capacity. The actual capacity increase will vary based on acid gas composition per Fig. 1. However the analysis is quite representative of the economic impact which is a savings in the mid 40% range across a wide range of SRU capacity. Applying a credit for N_2 offtake makes the savings 68% and it may be noted that the ASU can be designed to produce even more nitrogen than shown in Cases 3a and 3b. Note also that this analysis does not include the opex for the SRUs which would further tilt the equation towards O_2-based SRUs.

Some additional independent analysis on this topic performed by companies supplying industrial gases and/or oxygen enrichment technology is provided below:

An Air Products and GAA analysis comparing two scenarios (Table 4): four 400 t/d SRUs (one spare) versus three 300 t/d SRUs operating at 400 t/d with oxygen enrichment and an ASU. The analysis shows a capex savings of $90 million and some opex savings.

Fluor has done an analysis (Table 5) comparing four air-based SRUs with total capacity of 5,000 t/d versus two O_2-based SRUs with the same total capacity. They show a NPV savings of 40% for the O_2 based scenario.

WorleyParsons and Linde have done a detailed analysis recently comparing the costs of a new-build 1,000 t/d air-based and O_2-based SRU for acid gas strength ranging from 50% to 80% and show that the NPV savings for the O_2-based case are in the neighborhood of $90 to 140 million, increasing with lower acid gas strength.

Conclusions

In summary, all of these analyses show the immense economic superiority of O_2-based SRUs. Interestingly, the benefits are even greater for lower acid gas strengths as in gas plants where, ironically, oxygen enrichment has the least footprint at present. Finally, an oxygen plant is much more versatile than a SRU since its product can be used for other applications, e.g. enrichment of FCCs, furnaces, wastewater plants and the manufacture of other chemicals/petrochemicals at proximate sites. A spare SRU on the other hand is limited to the important but still singular purpose of providing redundant sulphur recovery capacity. There have been a lot of advances in air separation technology both in scale and efficiency in the last few decades since the start of SRU oxygen enrichment and this is manifested in the increasingly better economics offered by this configuration across the entire spectrum of feed size and compositions. ■

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Sulzer vertical sulphuric acid pump for your industry application.

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Sulzer offers a complete range of horizontal and vertical pumps, tailor-made to satisfy the customer specifications for sulphuric acid and molten sulphur plants. Advanced design and materials, a wide range of shaft seals and sealing systems, and the overall operating efficiency ensure a reliable process and a maintenance-free operation.

To learn what you can gain from our partnership, visit www.sulzer.com

Visit us at ACHEMA'18 in Frankfurt, Germany booth #8.0 A71.

SULZER

API 610 compliant Lewis® sulphur pumps

The new Lewis® product line includes a vertical type sulphur pump designed for API 610 service. **H. McKinnon** and **S. Race** of Weir Minerals detail the differences between conventional vertical sulphur pumps and those that meet API 610 requirements.

Weir Minerals has been manufacturing vertical sump pumps for molten sulphur applications since the 1940s. For many years, all that was required for their use in chemical, oil and gas plants was a standard sulphur duty pump. However, in the year 2000 it became necessary for sulphur pumps to be built to more stringent specifications as set forth in the API 610 standards. These standards, regulated by the American Petroleum Institute, cover all centrifugal pumps in the petroleum, heavy duty chemical and gas industry services.

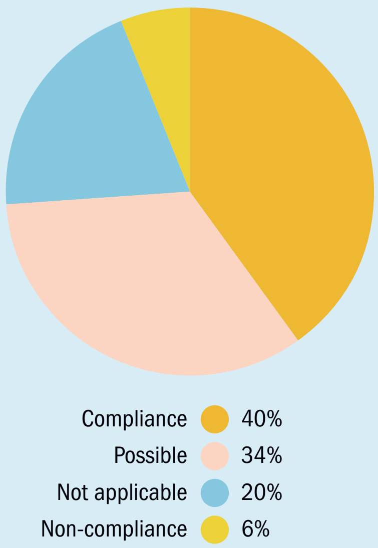
In response to these new standards, Weir Minerals added enhanced construction features and began to offer various tests and inspections that enabled it to respond to its customer's needs. In 2015, Weir Minerals reviewed the most recent API 610 11th edition specifications against the products that it had upgraded for the oil and gas industries. An extensive gap analysis was performed to identify what design changes were needed, the special test and inspection capabilities required, and the detailed documentation necessary to ensure that the company's vertical pump products complied with the API 610 standards.

As part of this process, Weir Minerals decided to take a closer look at whether total or near-total compliance was possible for its VS4 and VS5 type sulphur pump line from both an engineering and a business sense.

The decision process

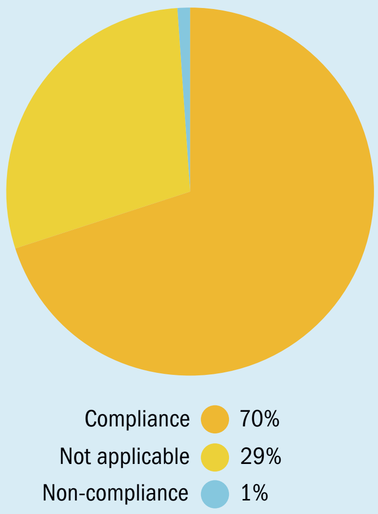
To facilitate a thorough decision-making and development process, a phase-gate team was formed to begin a clause by clause look at the entire document.

Fig. 1: **Feasibility team's intial assessment**



Source: Weir Minerals

Fig. 2: **Final assessment at completion of phase-gate process**



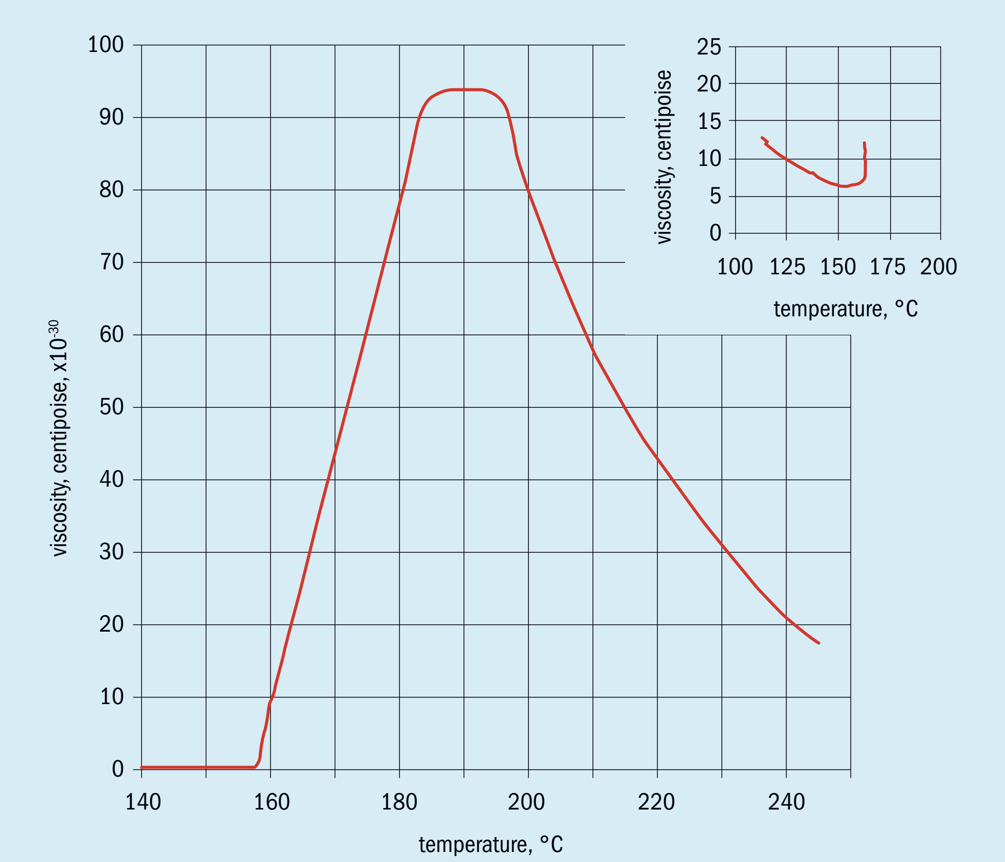
Source: Weir Minerals

During the feasibility phase, the Lewis® pump product line was reviewed against each individual clause of API 610 and placed to one of four categories: in compliance, not applicable to our pump, possible compliance with minor to moderate changes, and non-compliance. The team's initial assessment approximation was 40% compliance, 20% not applicable, 34% possible compliance, and 6% non-compliance (Fig. 1). After management reviewed the findings of the feasibility team, a decision was made to press on to the development phase and begin to work through the entire API document in detail, to assess more thoroughly the changes required for compliance, and their feasibility.

At the onset of the development phase, it was decided to limit the scope of the investigation process to a single pump model: the Lewis® 2VSHR sulphur pump was selected, as it is one of the most popular designs. If a decision to move forward with design changes was made, the findings in the development phase would be extended to other pump models at a later date. The development team, working for several months, concluded that the majority of non-conformances could be adhered to with simple changes, while others required some level of engineered change to the product line. The final level of compliance at the completion of the development phase is shown in Fig. 2.

Sulphur pumps, especially submerged sulphur pumps, are unique in several aspects; the ability to pump molten sulphur being one of them. Pure elemental molten sulphur displays a unique viscosity increase at around 160°C. As it goes

Fig. 3: **Viscosity of molten sulphur**



Source: Weir Minerals

through a phase change, the viscosity of molten sulphur increases almost exponentially with increasing temperature (see Fig. 3). The presence of entrained H_2S gas in the sulphur can mitigate the steepness of the viscosity increase to some degree. However, to control the viscosity, the pump is steam-jacketed and the steam pressure regulated so as to cool the sulphur internal to the pump and maintain the viscosity in the pumpable range. Submergence in molten sulphur provides unique challenges for pump maintenance as well: when sulphur cools it hardens to a concrete-like consistency, making it particularly difficult to remove from the pump during servicing. The slender cantilevered configuration of the VS4 and VS5 pumps also creates certain handling issues when laying the pump over on its side for maintenance. These and other features played into the specific clause-by-clause decisions regarding compliance, and are dealt with in more detail in the following sections.

To date there are three pump models that have been fully assessed against the phase gate team's findings and have had the necessary design changes incorporated: the Lewis® 2VSHR pump, 1.25VSH pump and 4MSHS pump. Additional Lewis® pump designs through size 6 sulphur pumps (6MSS) are planned for revision to the new API 610 line in the near future. Going forward, other larger sulphur pumps may be added to the API 610 product line as well. Some work has been done incorporating these changes in a Lewis® acid pump. Further consideration is also being given as to how API 610 might be applied to the sulphuric acid pump line; however these products require special consideration as API 610 does not cover pumps for sulphuric acid service.

Design changes

Many requirements in API 610 required little or no design changes to the Lewis® pumps. For example: the pressure casing stresses are already kept within the allowable range stated in clause 6.3: 0.67 times yield or 0.25 times the ultimate strength. Shaft critical speed is already a minimum of 1.2 times the maximum continuous speed. Nozzle loads at 2 times API Table 5 have been the design requirement for Lewis® pumps for some time. And bearing spacing for line shaft bearings already meets the requirements of clause 9.3.6.1.

The following are additional features of the new line of Lewis® API 610 pumps and within the requirements of API 610. Maximum impeller trims are selected to allow for a 5% minimum increase over the rated flow conditions by revising the impeller trim. The rated flow condition is established at 80% to 110% of BEP (best efficiency point), and the preferred operating range is set between 70% and 120% BEP. Cooling systems, including fans and/or cooling jackets for bearing housings, are used as required to maintain bearing temperatures. Higher speed pumps (>1,800 rpm) receive a heavy duty cover plate for enhanced vibration resistance.

Other API 610 requirements needed assessment and, where possible, a plan to change the design. In some cases, the method of manufacture needed to change. Testing methods and requirements, purchase parts, materials of construction, BOM (bill of materials) creation – in short almost every aspect of the pump fabrication process was affected by design changes intended to meet API 610 requirements.

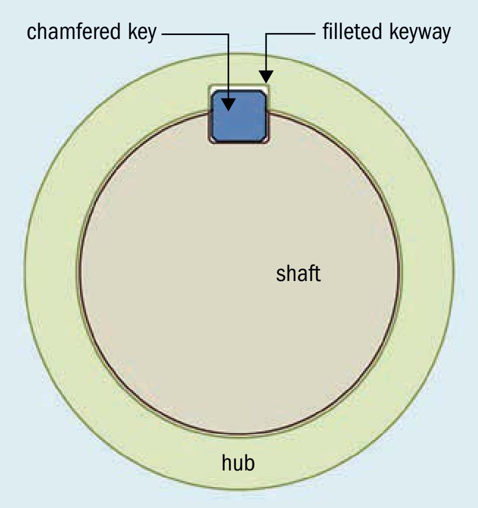
The following is a discussion of the significant changes to the pump line, as well as places where an exception was taken to the requirements of API 610.

Mechanical design changes

Keyway and key stock revised

API 610 requires keyways with filleted corners in accordance with ASME B17.1 (ISO 3117). Prior to the API 610 compliance effort, all keyways in Lewis® pumps were cut with square corners. Although squared-corner keyways never caused any issues due to the heavy shaft and hub designs, the change was viewed as a design improvement, as the shaft fatigue life was enhanced. However, changing all the keyways was not trivial; doing so meant, besides a cultural change, new machine tools and cutters.

Fig. 4: **Filleted keyway and key**



Source: Weir Minerals

Electro discharge machining (EDM) capability was added to cut the keyways and perform other machining operations. Key stock was revised to include a chamfer for proper fit in the new improved keyway configuration (Fig. 4). Rather than maintaining two keyway configurations, it was decided to revise the keyways and keys in all pump lines.

Rotor balancing

In the past, rotors were dynamically balanced to ISO 1940-1 grade 6.3 unless a higher grade was specified. The baseline impeller balance requirement in API 610 is grade 2.5. The G2.5 grade is the standard balance condition for all Lewis® API 610 pumps.

Discharge pipe weldment

For the Lewis® VS4 and VS5 pump configurations in API 610, the separate discharge pipe is considered as part of the casing (see Fig. 5). This meant that all welding on the inner process pipe had to meet ASME Sections V and VIII welding requirements. The jacketed configuration of this pipe presented a challenge; the inner pipe had always been welded to the flanges by access to the inner diameter only. Because of this limited access, full penetration was not possible, nor was any inspection of the backside of the weld. To comply with the API 610 requirement, the outer jacket was revised to a two-piece telescoping design. The weld design was brought into conformity with the boiler and pressure vessel code, and weld inspection was facilitated by the change.

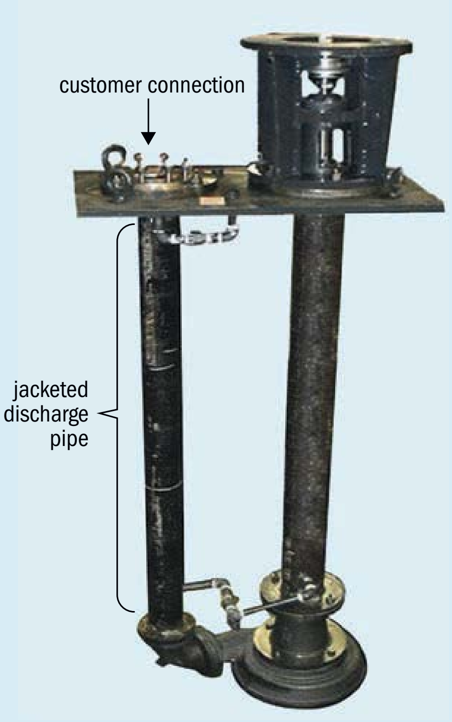
Ball bearing housing

API requires steel ball bearing housings. Typical Lewis® pump bearing housings are cast iron, necessitating some redesign. In some cases the new Lewis® API 610 pump ball bearing housing casting dies were modified to accommodate a steel shrink rate, other bearing housings were converted to a steel weldment. The stuffing box and gland follower were also changed to steel castings.

Ball bearings

Ball bearings required an upgrade as well in the new pump design. API 610 clause 6.10.1.4 lists several specific requirements for ball bearings: 7000 series bearings of a paired, single-row design, with 40°contact angle. They also must have machined brass cages. In addition, bearing life calculations are to be in accordance with ISO 281. Bearings are to have a minimum L10 life of 25,000 continuous hours at the rated

Fig. 5: **Jacketed discharge pipe considered as part of casing**



Source: Weir Minerals

conditions, and 16,000 hours at maximum radial and thrust loads, all at rated speed (the maximum load condition always corresponds to shutoff, as API 610 requires a 10% rise in head from the rated to shutoff conditions – see Hydraulics Changes).

This necessitated a change to all Weir Minerals ball bearings and a revision to the life-calculation methods as well. In some cases the bearing changes were significant enough to force a change to the bearing housings.

Hydraulics changes

Lewis® vertical sulphur pumps were originally designed to be run in a single-pump pit configuration. Since parallel operation was uncommon, the hydraulic designs were optimised for efficiency rather than curve stability. API 610 includes a strict requirement for continuously-rising curves regardless of application. In order to satisfy this requirement fully, new impeller geometries were designed to work in the original casings: adjusting the vane profiles and the number of vanes while maintaining the original shroud profile produced stable pump curves with a minimal investment in tooling (Fig. 6). Functional prototypes were produced from the correct alloys using a 3D-printed sand mould to accelerate validation.

Material changes

Wear parts such as wear rings, bearings and journals are made of hardened 12% chrome steel for S-5 and S-6 material classes, as specified in API 610 Annex H.1. Lewis® proprietary alloys are still available for wear and other parts, but their use requires a waiver from the customer releasing Weir Minerals from disclosure regarding the proprietary materials.

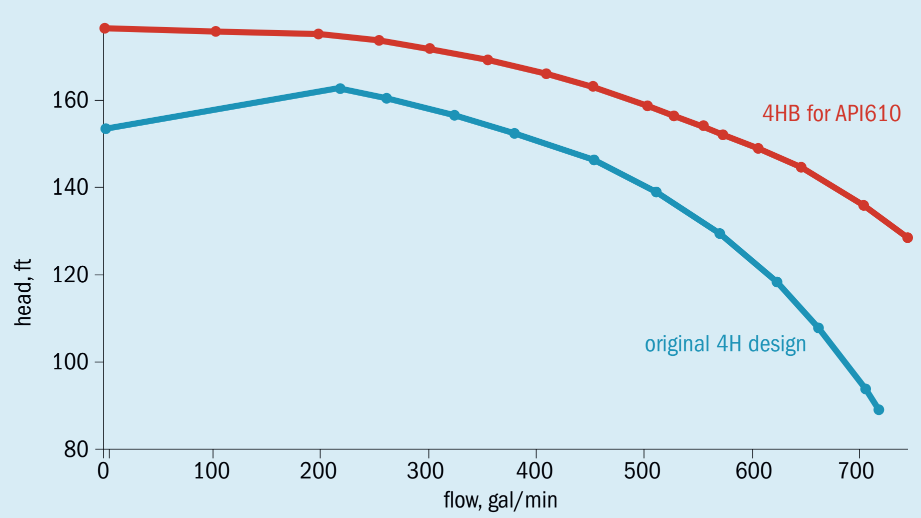
Reduced hardness materials according to NACE MR0103 or MR0175 are also available as the application dictates, and upon request.

API requirements excepted

Of the over 500 clauses of API 610, five were deemed as not acceptable in all cases:

1. Clean-out connections to be provided in steam jackets so entire jacket system can be cleaned out mechanically.

Fig. 6: **Impact of impeller geometry changes**



Source: Weir Minerals

Fig. 7: **Lewis® VS4 pump cross section showing steam jackets and crossovers**

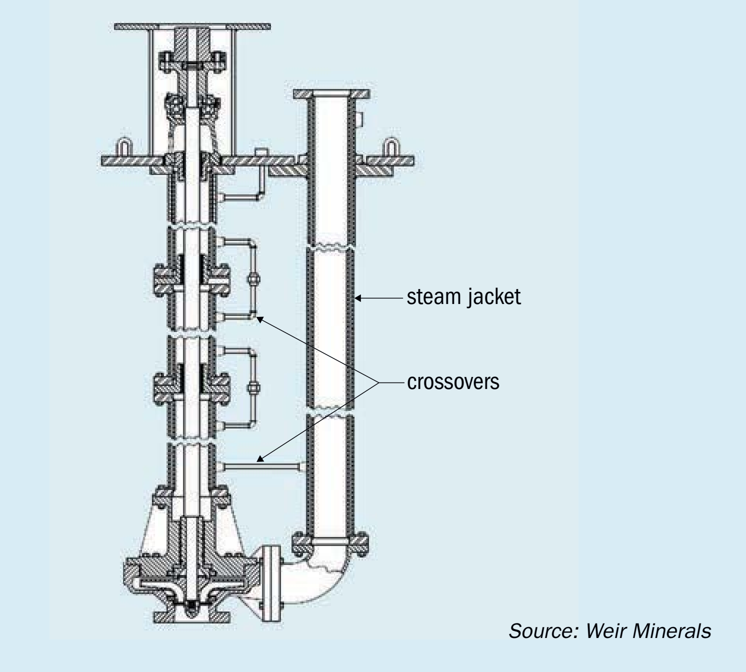
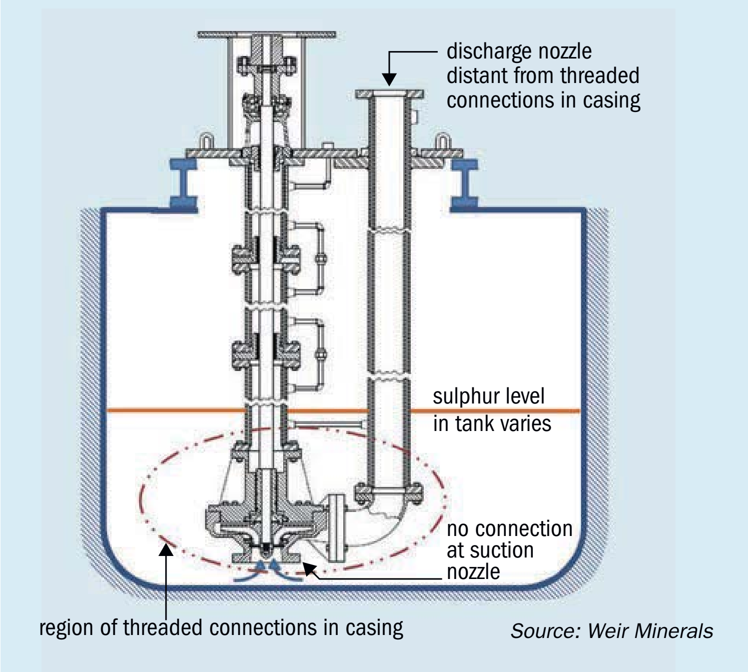


Fig. 8: **Lewis® pump installation showing proximity of nozzle connections**



2. Minimum material thickness around threaded joints in the casing to be equal to half the nominal bolt diameter plus the corrosion allowance.
3. Wear rings, in addition to interference fit, are to be retained by mechanical means: either pins, screws or tack welding are required in addition to the press fit.
4. Minimum wear ring clearances as prescribed by Table 6 of API 610.
5. Lift lugs at the cover plate required to lift pump as well as driver.

There are specific objections to each of these exceptions:

Clean-out connections in steam jackets: Clean-outs as described by API 610 can be problematic since the steam jackets for Lewis® VS4 and VS5 sulphur pumps are submerged in molten sulphur. Separable mechanical connections must be kept to a minimum below the cover plate and are deemed a risk. The steam jackets are welded to the boot and discharge columns to minimize the possibility of sulphur intrusion into the jacket, or conversely, water getting into the sulphur. Therefore, only the crossover pipes have a separable union, which can be used to flush and blow the steam jacket out (see Fig. 7).

Material thickness around threaded connections in casings: Casings for Lewis® sulphur pumps are most often made from the same casting designs as the sulphuric acid pumps. These casings are therefore extremely robust and heavy. In addition, on Lewis® VS4 and VS5 pumps, there is no piping connection at the suction nozzle (see Fig. 8). Consequently there are no suction nozzle loads on the casing; the suction end of the pump just hangs free at or near the bottom of the tank. Likewise the discharge-nozzle loads are applied at the end of the discharge pipe, far from the threaded connections in the casing. This combination of low loads and heavy construction invalidate the need for extra material around threaded connections.

Secondary mechanical means of retaining wear rings: Sulphur, when cooled, becomes a hard tough layer inside the sulphur pump, making disassembly difficult (see Fig. 9). Removal of the wear rings is already a tough job, and adding a retainer that is buried under a layer of hardened sulphur is not helpful to the maintenance of the pump. The Lewis® pump wear ring design uses an interference-fit and has not experienced issues with looseness; therefore, a secondary retainer is seen as a costly, cumbersome and unnecessary requirement (Fig. 10).

Minimum wear-ring clearance: API 610 stipulates maximum clearances for impeller wear rings to minimize internal leakage and improve efficiency. Lewis® vertical sulphur pumps use the sulphur to lubricate the line shaft bearings, the flow of which is regulated by the upper wear ring gap. As such, it is not possible to comply with the directive in API 610 without adding a costly direct injection system to the pump. The long service history of Lewis® pumps proves that this system is only necessary and beneficial in a limited number of applications as the impact of wider ring clearances on overall pump efficiency is minimal compared to the added cost.

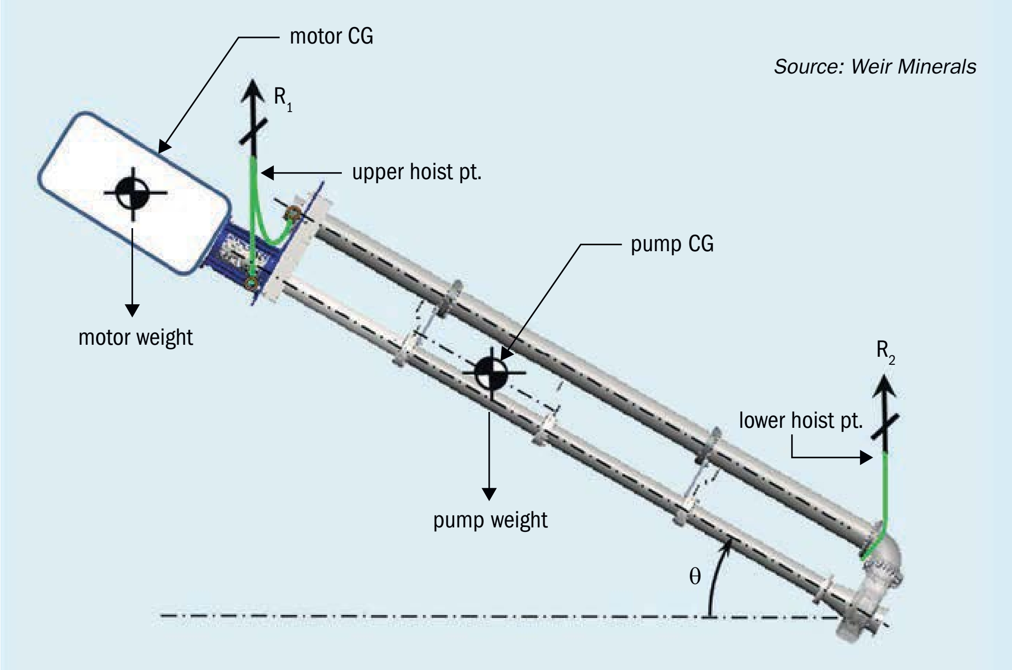
Lift lugs used for lifting pump and driver together: Lewis® VS4 pumps are often long, and require specific lifting and handling tech-

Fig. 9: Lewis® sulphur pump after removal from service, showing a heavy layer of sulphur under the suction head.

Fig. 10: Lewis® sulphur pump after cleanup – sulphur deposits still remain.

1 47 2 48 3 49 4 50 5 51 6 52 7 53 8 54 9 55 10 56 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46

Fig. 12: **Pump hoisting diagram, showing the process to lower the Lewis® sulphur pump**

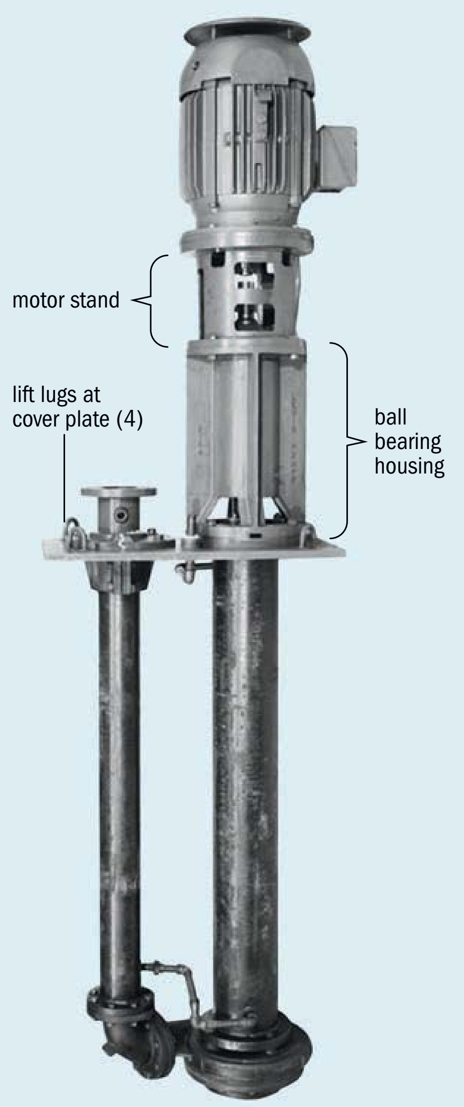


niques. Some sulphur pumps exceed 20 feet in length, but a common length is 10 to 12 feet, still a slender and long pump. By comparison, Lewis® VS5 pumps are relatively short, yet as is evident in Fig. 11, the motor is a long way from the lift lugs, and farther still from the pump's centre of gravity.

In general, when Lewis® sulphur pumps are lifted out of the tank, they come out vertically with the pump hanging straight down. If the motor is too heavy the load will be unstable and could invert. However, during most maintenance actions the pumps must be laid out horizontally. This is accomplished with the use of two cranes or hoists as can be seen in Fig. 12; the pumps are rotated to a horizontal orientation and placed in a cradle or on stands for disassembly. During such an operation, and with the motor still attached (which is frequently heavier than the pump), handling can be particularly precarious: if the motor is heavy enough, the load at the lower hoist point can reverse. Often these pumps are sold without a motor, so the motor weight isn't even known.

For this reason, lifting the pump with the motor or driver attached is unsafe and requires it to be removed prior to the hoisting of the pump.

Fig. 11: **Lewis® VS5 sulphur pump showing proximity of motor to pump and lift lugs**



Source: Weir Minerals

Testing changes

Performance testing

Weir Minerals recently built a state-of-the-art deep pump test pit, fully instrumented, to complete performance tests on fully assembled pumps. This advanced deep pump test pit enables Weir Minerals to accurately measure pump head capacity, power and NPSH, pump vibration, bearing temperature rise, overall sound pressure levels and resonance, among other things.

API certification testing is carried out in the in-house test pit. All parameters are recorded using an automated data acquisition system that exceeds the requirements of API 610. Due to the high viscosity of sulphur, a minimum of 10 points are recorded rather than the five required by API in order to increase the accuracy of the viscosity correction calculation.

Resonance testing

API 610 requires a resonance test on the assembled pump and driver. This test had not been performed at the Lewis® pump manufacturing location prior to the API programme. By leveraging the existing data acquisition system, this capability was a simple addition that nonetheless has proven valuable above and beyond API certification testing.

Summary

Weir Minerals now offer standard Lewis® sulphur pumps, as well as API 610 compliant Lewis® sulphur pumps, VS4 and VS5 style pumps. The API 610 compliant pumps are available in any material of construction class listed in the specifications with S1, S6 and A8 being the more standard configurations.

The changes made to the original product line in order to accommodate the stringent API 610 standards are significant improvements and will help provide dependable service. In particular, the pump's preferred operating range is now set to between 70% and 120% BEP, and the 10% minimum rise to shutoff from the rated condition helps achieve the performance required by API 610. In addition, improved construction techniques such as the discharge pipe weldment and shaft fatigue improvement are employed to enhance the life and durability of an already robust line of pumps.

Acknowledgement

This article is based on the paper "API 610 pump development" by Hal McKinnon and Scott Race of Weir Minerals and was presented at the Sulphur 2017 Conference in Atlanta, 6-9 November 2017.

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46
47 48 49 50 51 52 53 54 55 56

Meeting sulphuric acid catalyst challenges

Catalyst suppliers continue to develop new improved sulphuric acid catalysts to meet current and future challenges. In this article we report on Topsoe's new VK-711 LEAP5™ catalyst, offering a superior carrier system and an optimised chemical composition for improved intrinsic activity; BASF's new shape Quattro catalyst, providing a step change in catalyst activity with limited increases in pressure drop; and DuPont's MECS® GEAR® catalyst, featuring a hexa-lobed ring structure that increases the void space between catalyst rings, decreasing pressure drop and improving dust handling.

Legislation limits for the emission of SO_2 have been steadily decreasing, and how these demands are met influence the profitability of sulphuric acid plants. In the early 2000s acid plant operators who needed to boost plant performance could only choose between adding more catalyst of the same type, or adding caesium-promoted catalyst to the final beds. If there was no room for additional catalyst, and caesium-promoted catalyst was already being used, the plant operator had little option other than implementing a costly revamp of the plant. In cases where resources were not available for a capital project, the only option available to plant operators was to reduce production rate to meet new emission legislation.

Sulphuric acid producers continue to look for ways to expand production rates and reduce emissions levels with increasing pressure from their downstream customers and local governments. It is the responsibility of catalyst suppliers to meet these needs and to work with sulphuric acid producers to identify ways to improve their operation.

Topsoe's new LEAP5™ catalyst

With the continued tightening of SO_2 emission limits, Topsoe set out to bring converter performance to the next level by further development of the LEAP5™ series for improved activity and for other applications and operating conditions. The development strategy was built on detailed knowledge gained through fundamental studies of the working sulphuric acid catalysts combined with extensive experimental laboratory work, reaction engineering modelling and industrial full-scale validation.

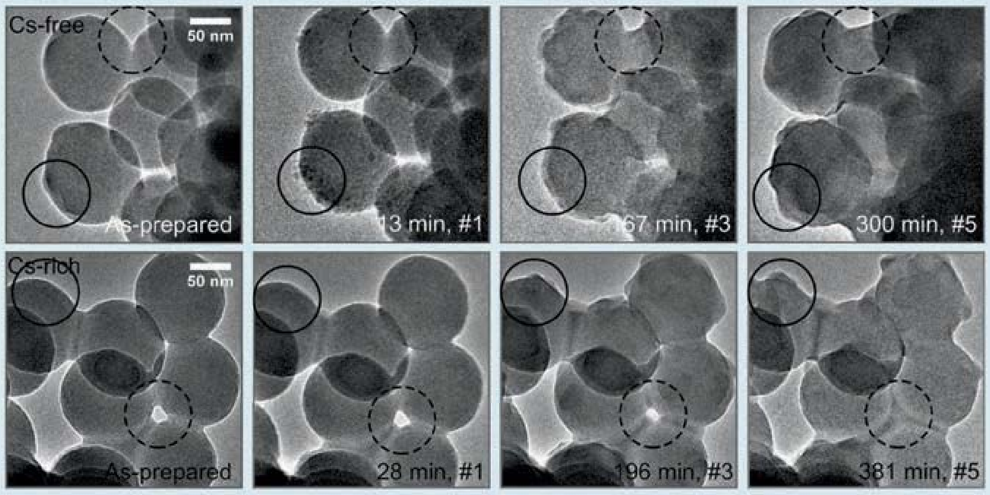
Commercial sulphuric acid catalysts are based on V_2O_5 dissolved in alkali-metal pyrosulphates on an inactive porous silica support. As catalysts are dynamic systems, which interact to a great extent with the local environment in which they operate, in-situ studies at relevant temperatures, pressure and gas composition are necessary to get a true picture of the working catalyst. This is particularly important for the vanadium-based sulphuric acid catalysts for which the active phase is a liquid that may account for about one third of the catalyst mass – a supported liquid phase (SLP) catalyst.

Three major interactions with the reaction environment should be mentioned as they can have a significant effect on catalyst performance. The catalysts have a significant absorption/desorption capacity for sulphur oxides (up to 10% of the catalyst weight) due to reaction between alkali metal sulphates in the catalyst and SO_2/SO_3 in the process gas. Secondly, an equilibrium exists between vanadium (V) and vanadium (IV) compounds in the melt. The degree of reduction to inactive vanadium (IV) is higher at low temperature and high SO_2 partial pressure, and it also depends on the liquid dispersion on the support. Furthermore, at temperatures below about 500°C some vanadium (IV) compound precipitates and gradually depletes the melt of active vanadium (V) when the temperature is lowered. Thirdly, the dispersion of the catalytic melt and consequently the available internal catalytic surface area depends on the reaction conditions and carrier morphology.

In order to better understand the details of the reaction mechanism and from that knowledge be able to rationally design new, improved catalysts, Topsoe has in recent years introduced new advanced in-situ techniques including Raman and high-resolution transmission electron microscopy to directly resolve the dynamic state of catalyst samples interacting with an SO_2/O_2/SO_3 gas mixture at temperatures from room temperature up to 600°C. These techniques have provided unprecedented insight into sulphuric acid catalysis[1-3].

An example of an in-situ TEM study is shown in Fig. 1. Model catalysts containing V_2O_5 and sulphates of either K (denoted Cs-free) or K+Cs (denoted Cs-rich) on 100 nm SiO_2 spheres were placed in the microscope (image denoted 'as prepared'). Subsequently, the catalysts were exposed to 10 mbar total pressure of 50% SO_2 and 50% O_2 at 450°C, and the dynamic changes of the catalysts were followed as a function of time. For the Cs-free sample, the vanadia phase in convex regions of the silica first transforms into smaller particles with a darker contrast and width of ca. 10 nm. Subsequently, these particles transform into more extended and faceted structures indicating that some areas of the convex surface develop crystalline character. In concave regions at the interstitial space between neighbouring silica particles, a molten vanadia phase seems to accumulate as this lowers the melt's surface energy. A similar behaviour is observed for the Cs-rich catalyst sample. However,

Fig. 1: **Time resolved TEM image of Cs-free (upper row) and Cs-rich (lower row) model catalysts at operating conditions**



Note: Solid circles outline convex regions, dashed circles outline concave regions

Source: American Chemical Society

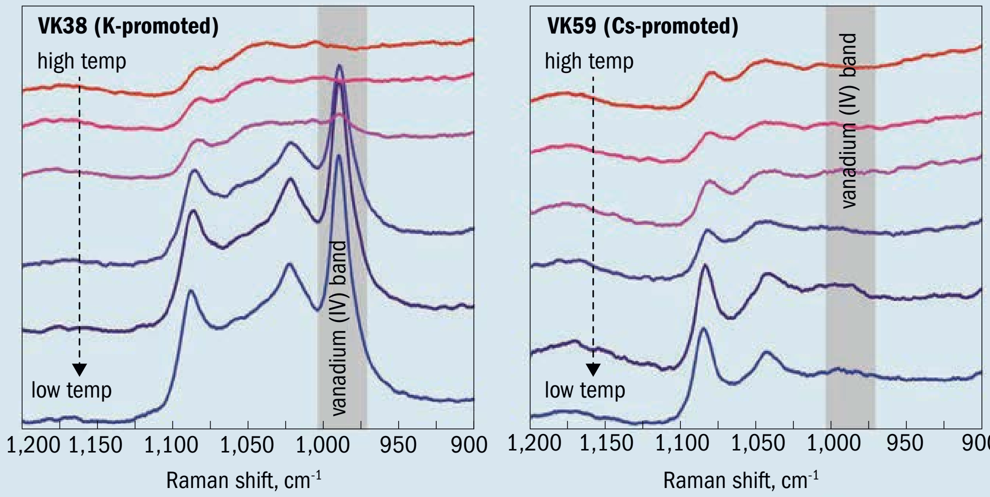
for the Cs-rich samples the transient particle formation in the initial stage was not observed in any of the monitored areas, and the extended facets on the convex surfaces tend to restructure with time to shorter and more compact features. Topsoe's studies also reveal that a molten phase emerges in the Cs-rich catalyst at lower temperatures than in the Cs-free.

In order to couple the physical transformations observed in the TEM with the chemical transformations taking place in the operating sulphuric acid catalyst, Topsoe has used an advanced operando Raman setup where both the chemical composition can be studied by Raman spectroscopy and the sample observed visually[3]. As an example of how the Raman has been used to gain better understanding of how the catalyst system behaves as conditions vary, or catalyst formulation is changed, see Raman spectra for K-promoted VK38, and Cs-promoted VK59 in Fig. 2.

In the left graph in Fig. 2, it is apparent how peaks associated with vanadium (IV) appear, and grow, in the Raman spectra for the K-promoted VK38 as temperature is reduced from close to 500°C to below 400°C. When looking at spectra for a Cs-promoted VK59 in the right graph, as temperature is changed in the same way, one will notice that no significant peaks appear in the same range. The lack of peaks in this area associated with vanadium (IV) species suggests that more vanadium stays and is available as active vanadium (V) species. Operando Raman can in this way help identify optimal formulations for a target set of conditions, such as low temperatures or high SO_3 content.

The fundamental knowledge gained through the in-situ studies on the interaction between carrier morphology and active phase combined with the observed details of promoter action at low operating temperatures has enabled Topsoe to design a superior carrier system and an optimised chemical composition for improved intrinsic activity in a new catalyst in the LEAP5™ series called VK-711 LEAP5™.

Fig. 2: **Raman spectra of K and Cs-promoted catalyst when going from high to low temperature, showing peaks associated with the formation of vanadium (IV) in the K-promoted, but not the Cs-promoted catalyst**



Source: Topsoe

VK-711 LEAP5™

The new VK-711 LEAP5™ is the second catalyst in the LEAP5™ series and builds upon the knowledge gained in the fundamental studies and the industrial experienced gained with VK-701 LEAP5™. The extra activity offered by this new technology can help operators overcome a number of different issues which could originally only be countered by a full scale revamp of the plant, such as:

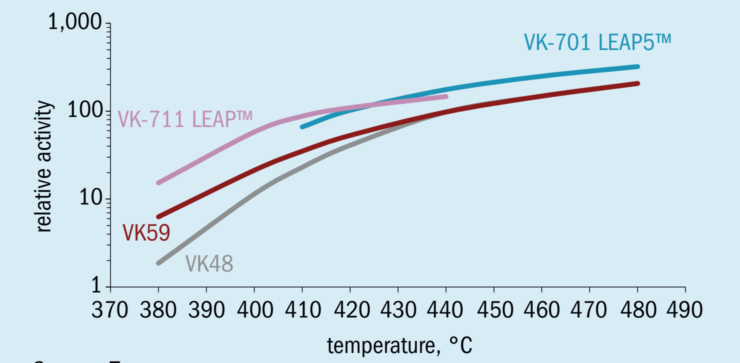
- decrease SO_2 emission;
- reduce scrubber chemical consumption;
- increase acid production or gas treatment capacity;
- increase oleum production;
- reduced plant pressure drop through enabling higher gas strength.

The activity of the VK-711 LEAP5™ as a function of temperature at high conversion in a feed gas with 10% SO_2 and 10% O_2 is compared to other Topsoe products, VK48, VK59 and VK-701 LEAP5™ in Fig. 3.

Fig. 3 shows that the new VK-711 LEAP5™ offers a step change in activity compared to a caesium-promoted VK59. This is true not only at the lower temperatures, but also at higher temperature, where normal caesium-promoted catalyst used at these conditions offers no advantage over standard potassium-promoted catalyst.

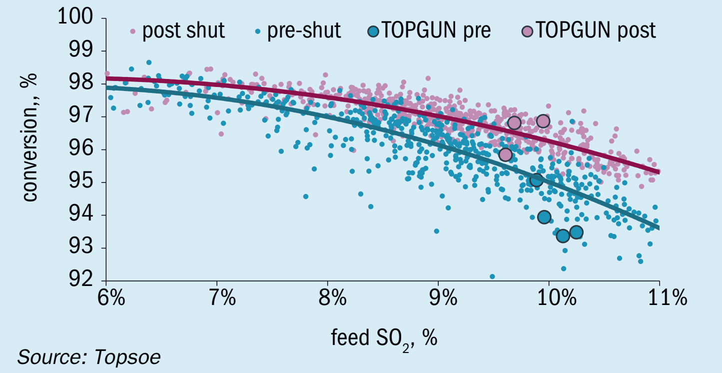
The extra activity offered by both catalyst utilising the LEAP5™ technology has often been employed by acid plant operators to meet new emission legislation, however others have used it to allow higher production capacity, or a combination of the two. In case the plant also produces oleum, and the economics favour oleum sales over sulphuric acid sales, LEAP5™ catalyst has also been employed to boost conversion before the oleum tower, and thereby oleum production. An overview of the different applications used for LEAP5™ catalyst so far is shown in Fig. 4.

Fig. 3: **Activity of the new VK-711 LEAP™ catalyst in comparison with VK48 and VK59**



Source: Topsoe

Fig. 5: **Conversion as a function of SO_2 strength, before and after installation of half a bed of VK-711 LEAP5™**



Source: Topsoe

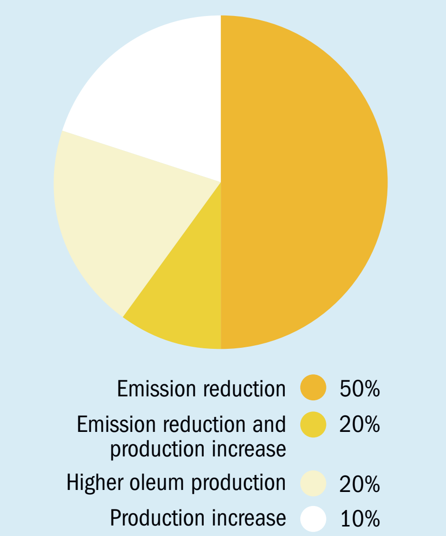
Industrial experience of increased production

The first acid plant operators have already benefitted from the advantages of the new VK-711 LEAP5™. One company, operating a three bed single absorption plant, faced challenges with the cost of delivering sulphur to the remote plant site. Due to the three bed single absorption layout, it was difficult to achieve high conversion, resulting in a significant part of the combusted sulphur being lost through the stack. The plant was already using caesium-promoted catalyst in the final bed, and although this catalyst was performing very well, the average conversion was limited to around 96% (see Table 1). With the new LEAP5™ catalyst, a new solution presented itself to reduce sulphur costs, without having to rebuild the plant. After different scenarios had been simulated and evaluated, it was decided to replace the top half of bed 3 with the new VK-711 LEAP5™.

After installation of half a bed of VK-711 LEAP5™, the plant experienced an average conversion improvement of just over 1 percentage point. Fig. 5 shows the conversion at different feed SO_2 levels before and after the installation.

The improvement by installing VK-711 LEAP5™ will depend on the feed gas strength. At lower SO_2 strength, lower activity will be needed to reach equilibrium, resulting in that improvement is lower than the average. At higher SO_2 strength on the other hand, the higher activity has greater effect, and the improvement is as high as 1.5 percentage points. The increase in conversion corresponds to a production increase of around 11,000 t/a with unchanged sulphur consumption. Through these sulphur savings, it is expected that this will offer a payback time of the catalyst within two years. In addition to the savings in sulphur cost, the improved conversion also results in decreased environmental impact of the plant.

Fig .4: **Motivation for using LEAP5™**



Source: Topsoe

Industrial experience of decreased emission

Another acid plant operator wanted to reduce the SO_2 emission from their single absorption plant. Alternatives considered were either to install a scrubber, or to improve the converter performance. Even before any changes were done, the plant and catalyst performed well with around 98.75% conversion. The operator decided to go with a catalyst solution, and the full last bed was eventually replaced with VK-711 LEAP5™, What convinced the plant operator was that conversion after installation of the VK-711 LEAP5™ would be 99.25%, at similar conditions. This corresponds to an emission decrease from around 1,000 to around 500 ppm (including some effect of more quench air). After start-up, the performance of the plant was assessed. Table 2 provides details of the recorded performance before and after installation of VK-711 LEAP5™, as well as the predicted performance.

As shown in Table 2, the improvement was greatly improved after installing the new VK-711 LEAP5™, and in line with what had been predicted. The very high conversion was achieved with an unchanged loading size of 257 L/t of acid, and despite a 17°C difference temperature difference over the cross-section of the final bed.

Table 1: **Data for plant with VK-711 LEAP5™**

SO_2 sources	**S burning and Cu smelting**
Configuration	3 bed single absorption
Design production, t/d	4,200
Feed gas strength, % SO_2	6-11
Average conversion, %	96
Loading size, L/t of acid	114 (design), 150 (average load)

Source: Topsoe

Table 2: **Performance before and after VK-711 LEAP5™**

	Prior to LEAP5™	**Post LEAP5™**
Production rate, % of prior	100	104
Feed gas strength, % SO_2	9.25	8.8
Inlet temperature, °C	419	393-410
Conversion, %	98.77	99.22
Emission, ppm	1004	573

Source: Topsoe

BASF Quattro catalyst

Through continuous improvement, BASF has achieved its goal of producing a step change in catalyst activity with limited increases in pressure drop to meet the needs of sulphuric acid producers. There are two primary ways to improve a sulphuric acid catalyst – recipe changes to the catalyst chemical structure and mechanical (shape) changes. Each of these options provide ways to meet the needs of the customer. BASF has focused on identifying the potential of various catalyst shapes. A natural first question on this journey is, "What makes a good catalyst?". The first factor is catalyst shape and its effects on pressure drop and geometric surface area. The second is the catalyst carrier. There needs to be a strong porous structure to allow access to active sites. The carrier also provides mechanical properties to the catalyst impacting crush strength and attrition. The final factor is the active compounds present in the catalyst and the number of active sites. These three factors impact the success of a catalyst and must be considered when looking to develop a new product.

The goal for a new BASF sulphuric acid catalyst can be summarised as:

- increased activity relative to the standard star ring 11 x 4 mm;
- little to no increase in pressure drop

To test this theory multiple star ring catalyst sizes were analysed and Fig. 6 shows the impact of catalyst size on this goal. CFD has shown that catalyst size, for the star ring shape, has a minimal effect on activity while strongly impacting pressure drop. Size adjustments alone will not meet the goal of a step change in catalyst activity.

Using a database of catalyst shapes the BASF research and development team used CFD to identify the best shape to meet the goal of increasing activity with limited increase in pressure drop. Fig. 7 shows three of the more promising results with the four-lobe shamrock shape offering about 30% increase in activity with close to a 10% increase in pressure drop. This shamrock shape was further researched leading to pilot production and commercial production trials; the resulting catalyst is called the Quattro.

Quattro development

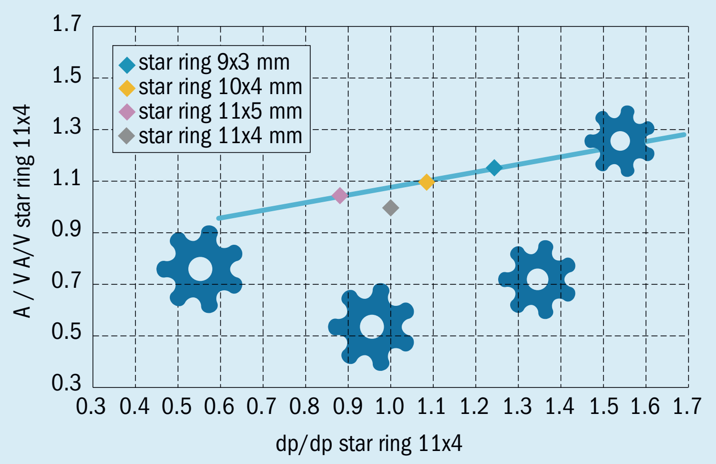
The development of the new Quattro catalyst took two years with production trials beginning in the first quarter of 2016. Due to impact for the customers, the Quattro was developed using the BASF caesium-promoted product line, 04-115, and was thus called 04-115Q. After completion of pilot and commercial production trials the BASF R&D team presented some positive results, see Table 3. The Quattro catalyst can meet the two goals of the project by achieving a step change in catalyst activity with minimal increase in pressure drop. The Quattro shape also provides improvements in cutting hardness and attrition %, resulting in longer active lifetime.

The Quattro catalyst is also flexible enough to operate across the whole temperature range of the sulphur dioxide oxidation reaction with consistent results above the reference, see Fig. 8.

Commercial trials

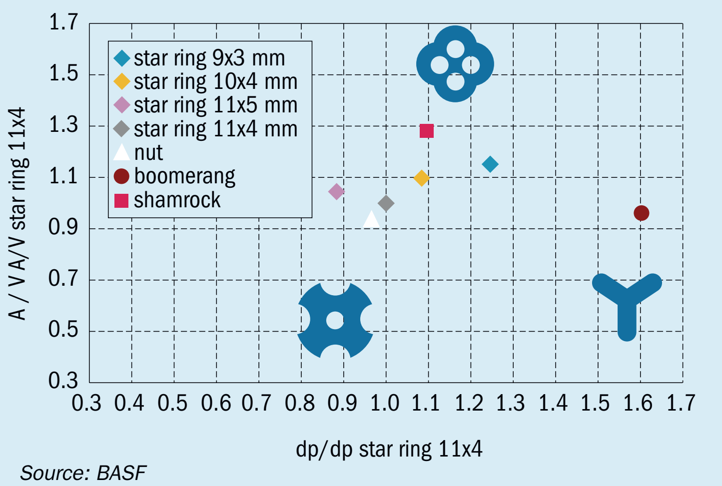
With the success of pilot and commercial production trials of the 04-115Q, the need for an external customer reference became apparent. A current customer approached BASF in the Spring of 2016 in need of a Cs catalyst

Fig. 6: **Impact of catalyst size**



Source: BASF

Fig. 7: **Impact of catalyst shape**



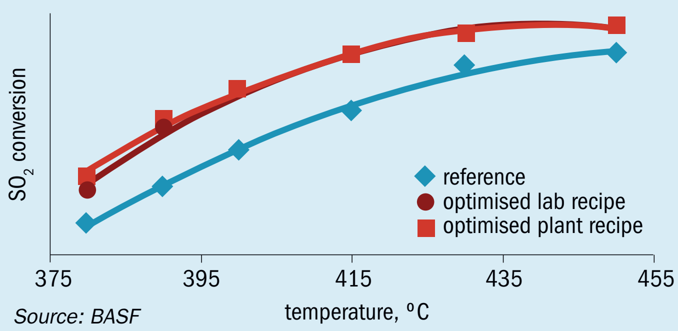
Source: BASF

Table 3: **Quattro R&D Results**

	Star ring	Quattro
Packing density, kg/m³	420	439
Relative geometric surface area, %	100	127
Pressure drop Re=100, %	100	110
Cutting hardness, N	86	101
Attrition, %	1.6	0.7

Source: BASF

Fig. 8: **Quattro performance across temperature range**



Source: BASF

Table 4: DOMO reference vs trial feed gas

	SO_2 (%)	O_2 (%)	O_2/SO_2 ratio	Capacity (t/d)	Conversion (%)
Reference	12.1	11.1	0.91	869	99.81
Trial	12.6	11.3	0.90	883	99.86

Source: BASF

Table 5: DOMO reference vs trial temperatures

	Bed 1	Bed 2	Bed 3	Bed 4	Bed 5
T inlet reference, °C	426	432	432	415	417
T inlet trial, °C	425	440	437	423	422
Delta T reference, °C	193	94	33	28	10
Delta T trial, °C	197	92	35	30	10

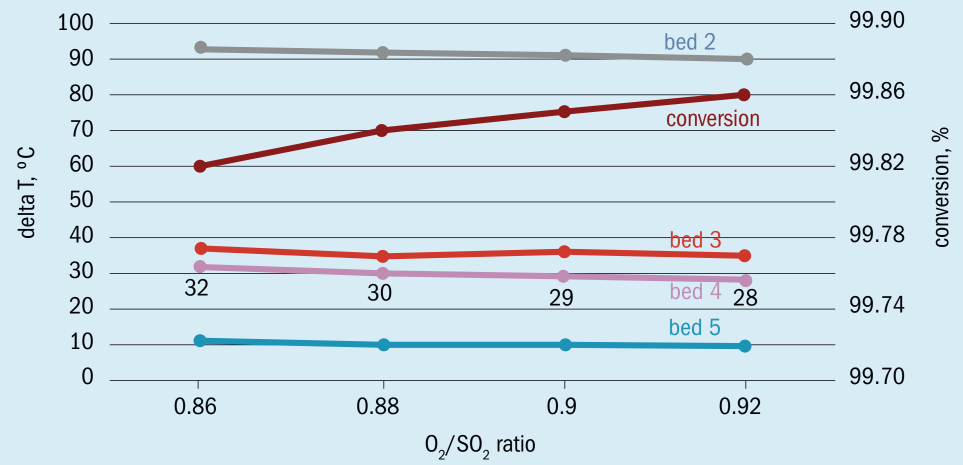
Source: BASF

Table 6: Comparison of performance testing October 2016 vs May 2017

	Bed 3	Bed 4	Bed 5	Capacity (t/d)
October 2016	90.64	99.54	99.86	883
May 2017	89.69	99.52	99.86	883

Source: BASF

Fig. 9: **Converter performance adjusting O_2/SO_2 ratio**



Source: BASF

changeout. Due to their current needs and bottlenecks, the 04-115Q was a good fit. DOMO Caproleuna in Leuna, Germany became the first external reference for the 04-115Q with installation in August of 2016.

The DOMO Caproleuna facility is a sulphur burning 3/2 double absorption unit in Leuna, Germany with a design capacity of 850 t/d. The feed gas has an O_2/SO_2 ratio of 0.9. The 04-115Q catalyst was installed in the fourth bed immediately downstream of the intermediate absorption tower. The trial plan was to test the activity of the new 04-115Q in the DOMO facility by increasing the SO_2 feed % while maintaining a constant O_2/SO_2 ratio and then adjusting the O_2/SO_2 ratio to test the flexibility of the catalyst.

After running for three months BASF conducted a Boss 100 conversion analysis to compare the success of the 04-115Q versus the standard 04-115 star ring shape previously in use by the plant. The results (Tables 4 and 5) showed two important points:

- The plant saw no increase in pressure drop in the fourth bed of the reactor where the Quattro catalyst was installed.
- The Quattro catalyst allowed for increased production rates over a flexible range of temperatures and O_2/SO_2 ratios with improved conversion.

Due to heat exchanger capacity, Table 5 shows increased inlet temperatures into beds 2 to 5. Due to the higher SO_2 content in the feed gas more energy was released from the exothermic reaction from SO_2 to SO_3 in the trial runs. The heat exchangers were run at maximum capacity but were not able to keep up with the gain in energy coming from the reaction. With properly sized heat exchangers for these operating conditions BASF believes this plant could see a production capacity increase of 6-8%.

BASF returned to the customer site in May of 2017 to test the performance of the Quattro catalyst again in comparison to the tests done in October of 2016. Due to the age of the catalyst in beds 1 to 3 the cumulative conversion through the first three beds had reduced by almost 1% versus the performance testing done earlier in the year. Due to the superior performance of the Quattro catalyst in bed 4, the cumulative conversion out of bed 4 remained the same as it was in October 2016 (see Table 6) allowing the overall conversion to remain at 99.86%. Due to this, BASF is confident that the plant capacity could be increased further.

BASF also wanted to better understand the performance of the 04-115Q catalyst over varying O_2/SO_2 ratios to provide improved flexibility to clients. The case study at DOMO kept a constant total gas flow rate of 70,000 Nm³/h, constant bed inlet temperatures, and varied the O_2/SO_2 ratio from 0.86 to 0.92. The results can be seen in Fig. 9.

The Quattro catalyst can be used to meet a variety of needs of a sulphuric acid producer:

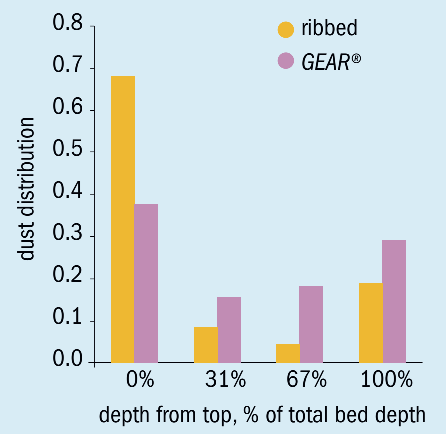
- Reduce emission levels: Higher active surface area results in better SO_2 conversion.
- Production capacity debottlenecking: Higher active surface area allows for increased production rates at historical conversion levels (5-8% improvement)
- Limited bed height: Higher active surface area allows for better performance in the same amount of space.
- Cost pressures on catalyst expense: About 30% less catalyst is required for the same conversion rates.

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46
47 48 49 50 51 52 53 54 55 56

Fig. 10: GEAR® hexa-lobed ring shape.

Fig. 11: Sleeves used in dust distribution study.

Fig. 12: **Comparison of dust distribution in catalyst beds after three years of operation**



Source: MECS

Table 7: **Bed pressure drop before and after replacement with GEAR® catalyst**

	Before catalyst replacement		After catalyst replacement	
Pass	**Catalyst**	**Pressure drop (mm wc)**	**Catalyst**	**Pressure drop (mm wc)**
1	ribbed with cap of ribbed caesium	340	GEAR® with cap of GEAR® caesium	95
2	ribbed	299	GEAR®	82
3	ribbed	336	GEAR®	95
4	ribbed with cap of ribbed caesium	136	ribbed with cap of ribbed caesium	163

Source: MECS

MECS® GEAR® catalyst

In 2011, DuPont introduced the MECS® GEAR® line of catalysts. GEAR® catalysts feature a hexa-lobed ring structure (Fig. 10). This structure increases the void space between catalyst rings, decreasing pressure drop and improving dust handling. Additionally, the improved formulation of GEAR® results in higher activity. Operating data from Mexicana de Cobre's La Caridad metallurgical sulphuric acid plant was used to study the ability of MECS® GEAR® catalyst to reduce pressure drop during the treatment of acid gas from copper smelters.

In 2015, Mexicana de Cobre elected to replace its existing ribbed catalyst with GEAR® catalyst in the first three passes of its catalytic converters at its La Caridad sulphuric acid plant. Operational data from the plant shows that the open structure of GEAR® significantly decreases pressure drop through the catalyst bed while improving upon overall conversion.

Impact of shape on dust distribution

During the operation of sulphuric acid plants, a fraction of the acid gas sent to the catalytic converter is composed of particulate matter. The primary sources of this particulate matter are the ash content in the raw material that is combusted to form SO_2 and dust in the unfiltered air of combustion. During the operation of sulphuric acid plants, the particulate matter is typically captured by the first pass in the catalytic converter. The accumulation of this dust results in increasing pressure drop through the pass, eventually requiring shutdown of the plant and catalyst screening once the pressure drop has exceeded the maximum allowable by the process blower. Frequent downtime and catalyst screening entail significant costs for acid producers.

As dust accumulates in the catalyst bed, it is important that it does not accumulate solely in the front of the bed, as this would lead to rapid build-up of flow restrictions in the void spaces between catalyst particles. Instead, it is preferable that dust accumulates throughout the entire catalyst bed in order to avoid a high level of flow restriction at any single depth of the bed. Proper catalyst-shape design can facilitate even dust distribution. Compared to standard ribbed ring catalysts, GEAR® catalyst demonstrates improved dust distribution. This is due to the unique hexa-lobed ring shape, which increases the void space in the catalyst bed, allowing dust to penetrate farther into the catalyst bed.

In order to demonstrate the superior dust handling capabilities of GEAR® catalyst, a test was performed at a MECS® sulphur-burning sulphuric acid plant, in which sleeves (see Fig. 11) containing about 200 litres of catalyst were inserted into a catalyst bed. After three years, the sleeves were removed from the bed, and the amount of dust was quantified at various bed depths. The results are shown in Fig. 12.

As shown in Fig. 12, nearly 70% of the dust accumulated in the top third of the sleeve containing ribbed catalyst. In comparison, a little less than 40% of the dust accumulated in the top third of the sleeve containing GEAR® catalyst with the remaining dust distributed on a fairly even basis across the bottom two thirds of the sleeve.

Performance data at Mexicana de Cobre

Mexicana de Cobre is part of Grupo Mexico. The La Caridad facility is located in Sonora, Mexico. Mexicana de Cobre operates two sulphuric acid plants at the La Caridad site, which treat acid gas produced during copper smelting. In 2015, Mexicana de Cobre's La Caridad sulphuric acid plant replaced the catalyst in the first three passes of one of their catalytic converters. Before replacement, all four catalyst passes contained ribbed catalysts, as shown in Table 7, with passes 1 and 4 including caps of caesium-containing ribbed catalyst to facilitate light-off. After replacement, the first three passes contained GEAR® catalyst, with the first pass including a cap of caesium-containing GEAR®. The fourth pass was not replaced, still containing ribbed catalyst with a cap of caesium-containing ribbed catalyst.

As shown in Table 7, before catalyst replacement, when each of the beds consisted of ribbed catalysts, the pressure drop

Mexicana de Cobre's La Caridad sulphuric acid plant.

in passes 1-4 was 340, 299, 336, and 136 mm wc, respectively. After catalyst replacement with GEAR® catalyst, the pressure drop in passes 1-3, while maintaining the same operating capacity and catalyst bed volumes, was 95, 82, and 95 mm wc, corresponding to pressure drop decreases of around 72% in each bed. The fourth pass, which was not replaced, showed a slight increase in pressure drop from 136 to 163 mm wc, or 20%. In addition to the improvements in pressure drop, the outlet concentration of SO_2 decreased from 635 ppm to 345 ppm. These results demonstrate the dramatic improvements in pressure drop resulting from the GEAR® shape. ■

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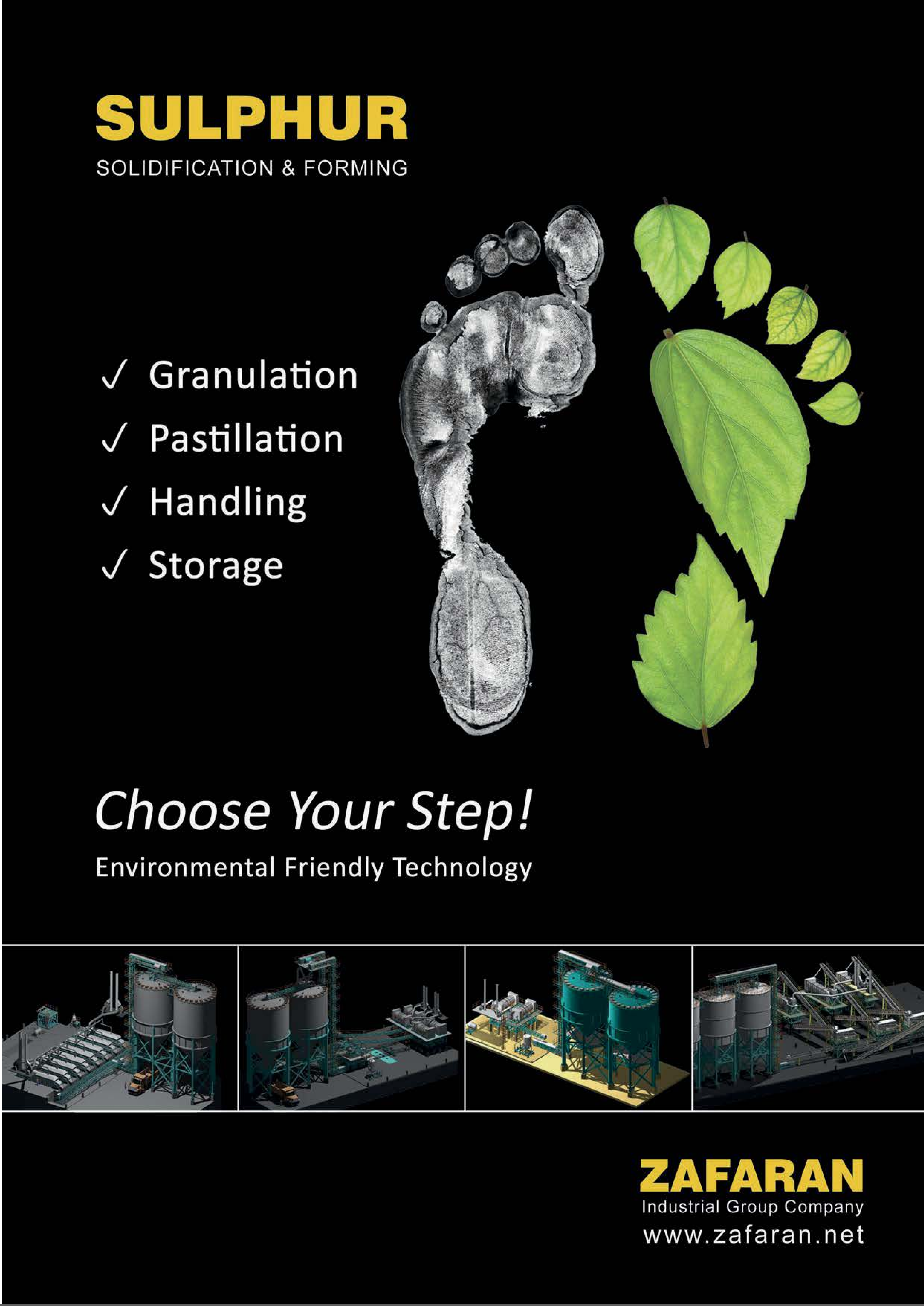
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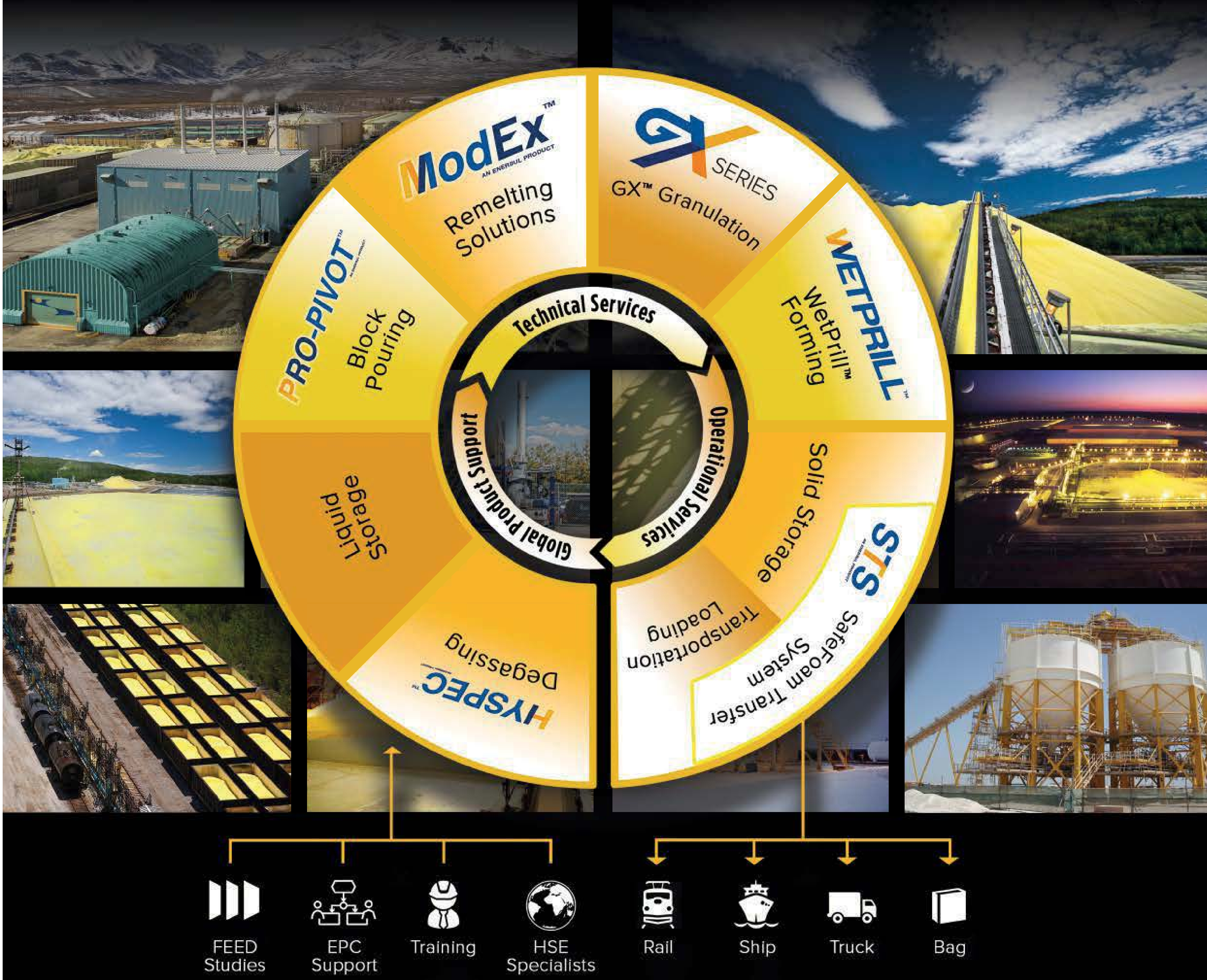

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