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The changing nickel market

Refineries after Covid

Sulphur recovery using WSA

New tail gas catalysts

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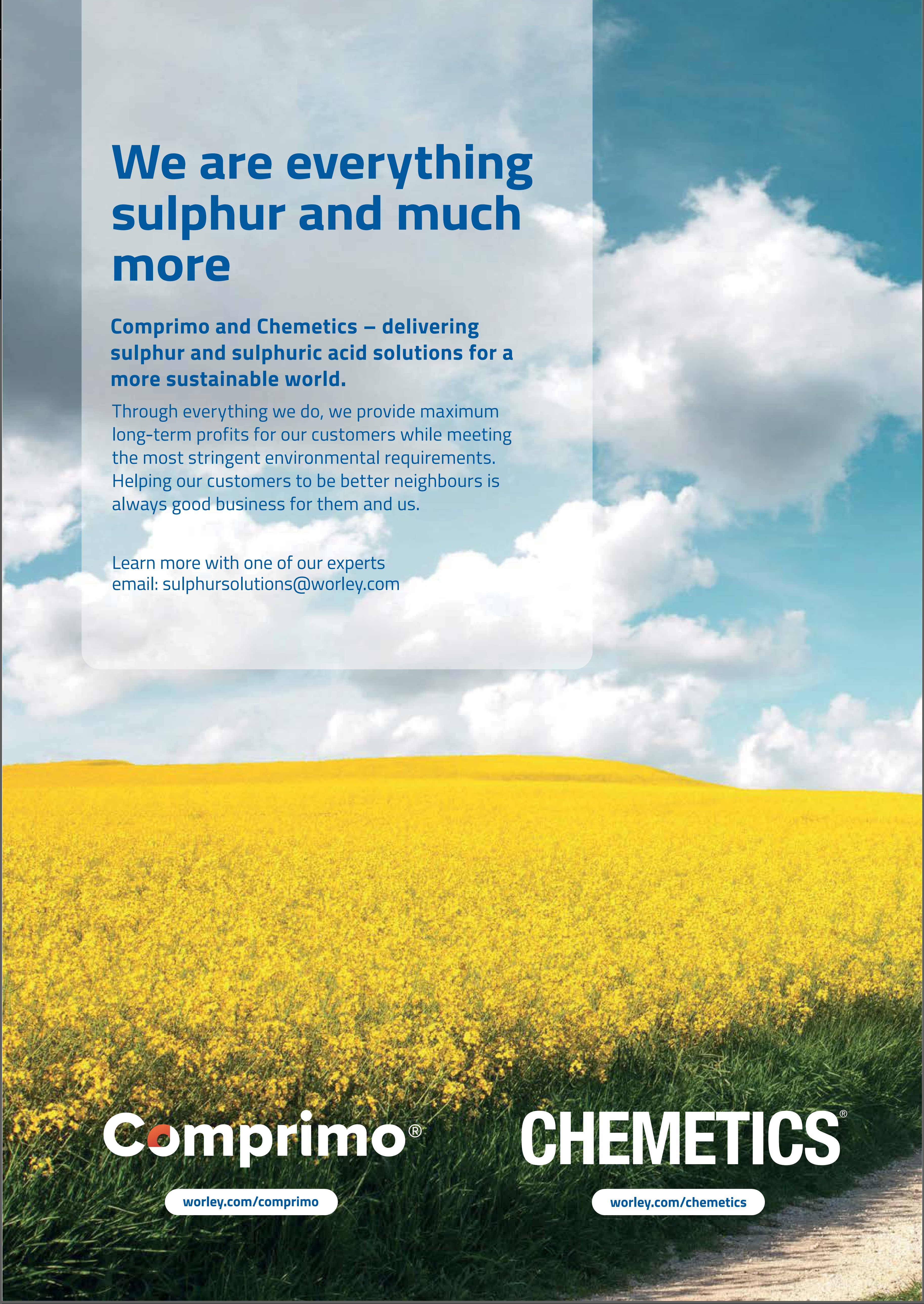

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Cover: *Electric vehicle charging.*
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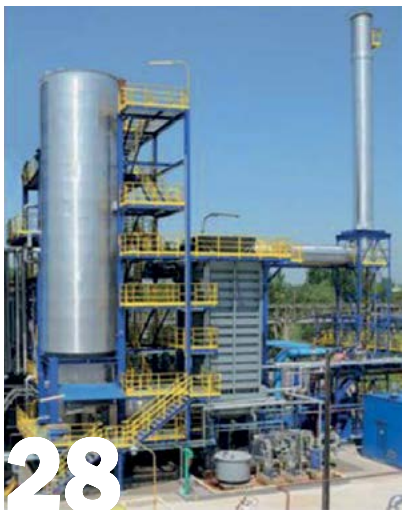

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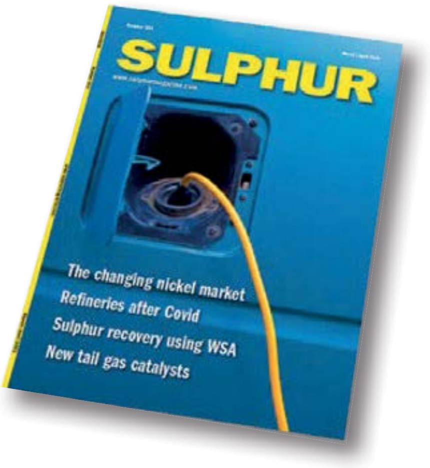
A rising tide



> Suddenly it's a good time to be a sulphur producer again.

Suddenly it's a good time to be a sulphur producer again. Sulphur prices started 2020 at a low point of \$40/t, a level not seen for a decade or more. However, after a slight recovery when the pandemic hit and refineries began reducing production, since August the market trend has been rapidly upwards, now approaching levels of \$200/t that have not been seen for a couple of years.

The story is one of tight supply and strong demand. On the supply side, the impact of the Covid pandemic and its associated lockdowns has been to reduce demand for fuels, especially aviation fuel, but also gasoline and diesel, and refineries as a consequence have been forced to cut back on production. In the US, refinery closures may have removed up to 600,000 t/a of sulphur production from the market. Europe has also seen refinery closures and drastically reduced run rates. Other, seasonal factors have also had an impact. For example, winter is traditionally a tighter time in sulphur markets, as the freezing of rivers in central Asia affects availability from Russia and Kazakhstan, and that should ease as warmer weather returns. Winter storm Uri in the US also closed much of the US Gulf Coast's refining capacity due to power outages.

Meanwhile, on the demand side, phosphate fertilizer demand for sulphur has held up during the pandemic, aside from some early and relatively short lived shutdowns in India and China. Overall sulphur demand was down in 2020 on 2019, but only by about 800,000 tonnes. Demand has been strong in Asia ahead of the spring application season. DAP prices have also been boosted by a spike in ammonia prices. The winter storm in the US which affected many Gulf Coast refineries also shut down a lot of ammonia production, and that happened at the same time as unplanned outages of ammonia plants in Trinidad and elsewhere. Strong phosphate pricing has helped support higher sulphur prices. There have also been restarts for sulphur burning metallurgical plants, including Ambatovy in Madagascar, and copper and nickel demand is strong as the global economy improves.

Because production is largely involuntary, the sulphur market is not one that is able to react quickly to rapid increases in demand. While there are stockpiles, they are not always accessible (e.g. in northern Alberta or central Asia). The past few months have seen accessible stocks fall – Chinese port stocks of sulphur have fallen dramatically, reducing by about 700,000 tonnes during 4Q 2020. This means that, provided demand continues to hold up, prices should stay high until new supply becomes available.

As global refining capacity recovers, so sulphur output should increase, particularly in the second half of 2021. There are also major new oil and gas projects which will generate large volumes of sulphur due to start up this year, including the long-delayed Barzan project in Qatar, new refineries in Kuwait, and the ramping up of gas plants in Saudi Arabia. Barzan and the Clean Fuels Project in Qatar between them represent 3 million t/a of new sulphur capacity, although any new project needs several months to actually achieve that. Likewise phosphate demand is seasonal, and high prices will tempt producers to keep producing, gradually easing the current run up of prices. For the time being, however, sulphur is very much back to being a co-product and not a by-product. ■

Richard Hands, Editor

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Price Trends

MARKET INSIGHT

Meena Chauhan, Head of Sulphur and Sulphuric Acid Research, Argus Media, assesses price trends and the market outlook for sulphur.

SULPHUR

Global sulphur prices have continued to escalate through the first quarter, accelerating at a rapid pace through to March. Delivered prices breached the $200/t c.fr mark in India at the time of writing while Middle East prices were set at over $50/t higher for March on the February level. The bullish tone towards the $200/t c.fr level left some buyers hesitant and retreating to the sidelines, but with expectations for firm demand and continued tight supply, prices are likely to rise higher before reaching a ceiling. Commodity markets are supporting demand from metals leaching projects with copper and nickel prices seeing a strong boost on the back of demand from key regions including China.

The lack of liquidity is expected to further propel prices in the near term and Middle East prices are edging closer to the $200/t f.o.b. mark. Middle East producer pricing for March 2021 was set at $183/t f.o.b. across the board. In the UAE, this reflected an increase of $55/t on February for ADNOC liftings to the Indian market. KPC/Kuwait raised the price by $58/t f.o.b. while the Qatar Sulphur Price was raised by $58/t.

The Chinese Lunar New Year period failed to bring a break to sulphur purchasing or the price run. The holiday period typically sees Chinese buyers step back from the market for several weeks, but a combination of factors limited the usual seasonal industry slowdown. Travel restrictions remained in place across many regions in China, understood to have led to some industries incentivising employees to continue working through February. High phosphate prices also contributed to the fertilizer sector maintaining higher than expected output levels. DAP operating rates sat at around 65-70% in February in Yunnan, 70% in Guizhou and 65-70% in Hubei. Demand for cargoes remained elevated, driving prices higher than expected.

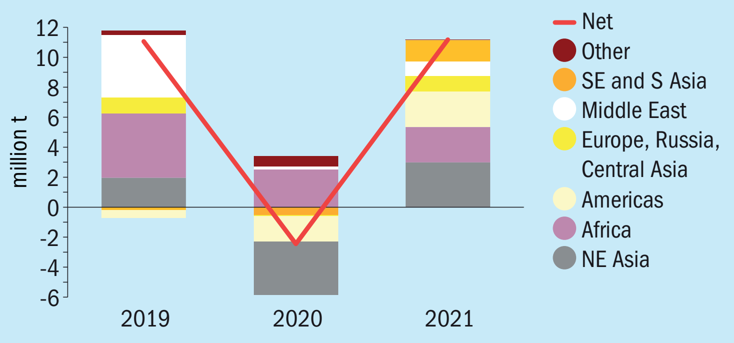
Rising freight rates have also added to the price rally since the start of the year, widening the spread between Middle East f.o.b. rates and China c.fr through January and February. Average China spot prices in February were $141/t cfr and $183/t c.fr on the high end, representing an month-on-month increase of $28/t and $43/t respectively. Prices at the start of March edged up further, at around $200/t c.fr for granular product, with further increases anticipated, on the assumption freight rates remain firm.

With the short term market balance so tight, a big question mark is around when and if new supply will emerge to ease availability issues. The two key projects in the short term that are awaited are the Clean Fuels Project (CFP) in Kuwait and Barzan in Qatar. Combined, these would add around 3.0 million t/a sulphur capacity to the market. The CFP has progressed with units being commissioned at the end of 2020. Sources have suggested all remaining units at Mina Abdullah should be commissioned by the end of the first quarter. In terms of sulphur volumes, Argus expects to see a gradual start up, owing to the disruption from Covid-19. Barzan meanwhile is still pegged for startup during 2021, but volumes are also likely to be added gradually.

Average Indian market prices have been assessed ahead of China during this price rally and the first market to hit $200/t c.fr in February. Domestic refiners in India have been operating at reduced rates, limited domestic sulphur availability, keeping import demand firm. At the start of March PPL purchased a cargo for March arrival to Paradip priced at $230/t c.fr with credit. The buyer has a scheduled turnaround planned for April lasting a month at its Paradip NPK/DAP plant. Meanwhile CIL bought a 40,000 tonne sulphur cargo priced at $230-$232/t c.fr for a two port discharge for March arrival. On the supply front, increased output is expected in the country. Hindustan Petroleum plans to add a new refinery at Barmer in Rajasthan by 2023 adding over 60kt/y sulphur capacity.

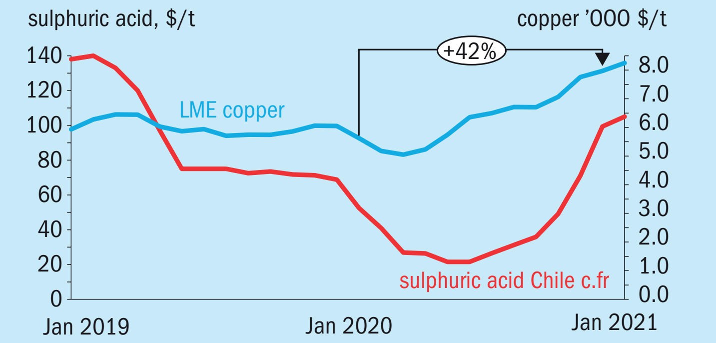
The restart of nickel operations at First Quantum's Ravensthorpe mine in Western Australia at the start of 2020 has boosted sulphur trade. Australian imports rose by 82% in 2020 on 2019 levels to just over 1 million tonnes, supported by increased demand from the ramp up of the project. Canadian sulphur made up 68% of deliveries and Qatar delivered 335,000 tonnes after an absence on this trade route in 2019. In 2020, First Quantum produced 13,000 t of nickel but expects the mine to produce 23-27,000 t in 2021 as operations complete ramping up. Favourable nickel prices continue to support leaching operations. LME nickel prices reached levels not seen since 2014 trading at $19,722/t in February before retreating back down to below $18,000/t. The medium term outlook for nickel based demand remains robust owing to the spate of leaching projects expected to come online in Indonesia, with several speculative projects in Australia. The projects are expected to include sulphur burners increasing sulphur import demand further.

Fig. 1: **Forecast sulphuric acid demand changes**



Source: Argus Media

Fig. 2: **Copper vs sulphuric acid prices 2019-2021**



Source: Argus Media

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SULPHURIC ACID

Global sulphuric acid prices have seen sharp increases, supported by strong market fundamentals. Continued tight supply, buoyant sulphur prices and strong copper prices have contributed to the run up in achievable prices. The bullish market sentiment is expected to continue in the short term, with no sign of a ceiling being reached. Further price rises were anticipated through March across the major benchmarks. Global demand is forecast to recover with rising commodity market prices and vaccine rollouts in some regions supporting acid consuming sectors. Following the net decline in growth in 2020 on the back of Covid-19 related disruption, Argus is forecasting a strong recovery of over 11.0 million t/a in 2021. On the supply side, smelter acid production was disrupted in 2020 by the Covid-19 fallout. A recovery is forecast in the short term as operating rates improve with 0.9mn t of acid supply growth currently anticipated. At the same time, turnarounds for 2021 are expected to lead to a loss of over 1.0 million tonnes acid. Increases on this are expected as further schedules emerge in the coming months. Delays to maintenances in 2020 came as a result of Covid-19 disrupting operations with lower than usual outages over the year.

Northwest European fob spot prices rose to $50-60/t f.o.b. in mid-February from $27-35/t f.o.b. a month earlier. Producers in the region have had little to no spot available in recent weeks but were receiving regular enquiries as consumer operating rates were strong. Sulphur burner acid availability is expected to remain tight in the short term with three sulphur burners expected to come offline in the coming months, tightening supply further. European smelter acid contracts for the first half of 2021 were assessed at €61-72/t c.fr. The top end of the range is up by €7/t on the previous contract price.

Negative prices ex-Northeast Asia were a market talking point in 2020 but in recent months prices have risen sharply. Tight availability and healthy spot interest has lifted major Asian prices. Japan/South Korean acid prices were assessed the same level as in Europe at $50-60/t f.o.b at the end of February. Meanwhile China spot prices were assessed at $70-80/t f.o.b., with the average price up by $60/t on the end of 2020. Domestic demand in China has been strong, with limited supply reported at sulphur burners.

Global demand for sulphuric acid for phosphoric acid production is expected to see growth in 2021 following the disruption in 2020. The pandemic slowed down economic activity and resulted in a downside to phosphate application during key planting schedules. For 2021, Argus expects demand from the phosphoric acid sector to see increases across almost every region, adding 4.5 million t/a of sulphuric acid consumption. Agricultural sectors showed the most resilience in comparison to other commodity markets in 2020. Most crop prices dropped in March/April but quickly recovered. The limited disruption stemmed from most governments declaring the food and agricultural sectors as essential. Agricultural trade has also been less affected than other goods as bulk shipping saw less disruption than other modes of transport.

On 24 November 2020, the US Department of Commerce issued a preliminary ruling on import duties for Russian and Moroccan phosphate shipments to the US. This followed US producer Mosaic's petition to introduce the duties. The duties have since been changed. For OCP duties increased slightly to 19.97% while tariffs on Russian product fell to 9.19% for Phosagro and 47.05% for Eurochem. The duties had deterred shipments to the US and led US phosphate prices to rise sharply relative to other global benchmarks with spreads reaching 5+ year highs. Domestic DAP/MAP production levels in the US were expected to shift in response and increased domestic phosphates production would have seen US sulphuric acid consumption rise. The US International Trade Commission's final ruling is scheduled for 25 March when the commission will decide whether to levy the revised duties.

On the copper front, Freeport McMoRan's Lone Star copper leach project in Arizona will continue to ramp up production through 2021 following a 2020 start up. The site will produce 91,000 t/a of copper at capacity. Based close to Freeport's Safford operations, the new copper mine and SX-EW plant will benefit from existing infrastructure, including the Safford sulphur burner. A plan is also in place to advance studies for a potential expansion and long term development options. ■

Price Indications

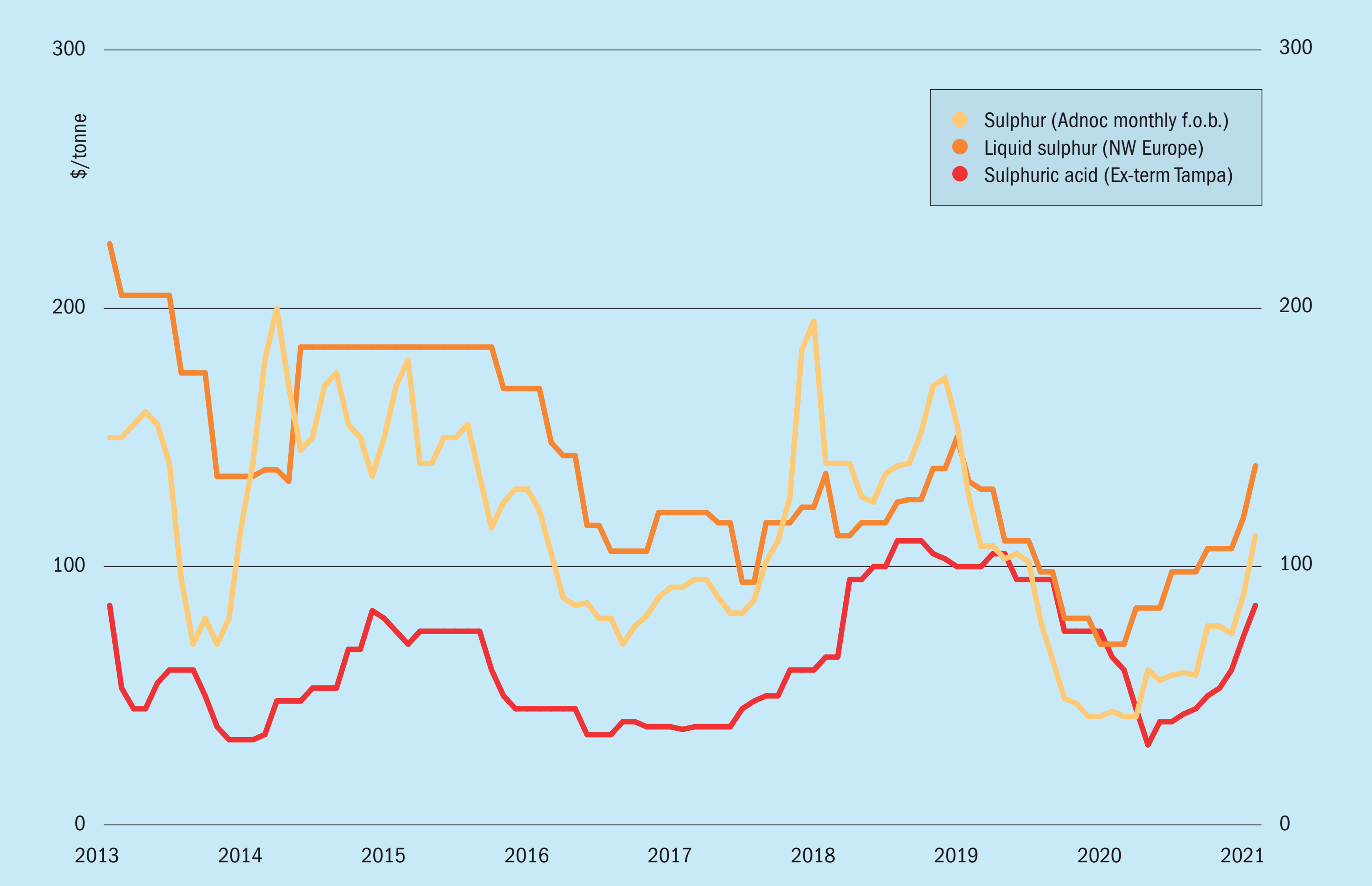
Table 1: **Recent sulphur prices, major markets**

Cash equivalent	**September**	**October**	**November**	**December**	**January**
Sulphur, bulk ($/t)					
Adnoc monthly contract	77	77	74	89	112
China c.fr spot	93	95	115	115	141
Liquid sulphur ($/t)					
Tampa f.o.b. contract	58	69	69	69	96
NW Europe c.fr	107	107	107	119	139
Sulphuric acid ($/t)					
US Gulf spot	50	53	60	73	83

Source: various

Market Outlook

Historical price trends $/tonne



Source: BCInsight

SULPHUR

- Restrictions and lockdowns across the globe have significantly reduced oil demand, impacting refinery run rates. This has aided in reducing sulphur liquidity, supporting the short term view.
- Speculative traders in China stepped in during Q4 2020, driving up prices ahead of the spring application season. China port inventories dropped in December, leading to a flurry of buying.
- Brazilian sulphur demand is expected to increase in the short term with rising phosphoric acid production. Sulphur imports are forecast to tick up by 0.3 million tonnes in 2021 in parallel to meet growing demand.
- Increasing phosphoric acid production at OCP's/Morocco operations in 2021 is expected to raise sulphur import demand. Supply from the Middle East will likely increase as a result.
- Global sulphur demand recovery is forecast in 2021 following erosion in 2020 from Covid-19 related disruption. The processed phosphates sector is expected to see strong growth in particular.
- Two Middle East projects are expected to start ramping up in 2021 – Barzan in Qatar and the Clean Fuels Project in Kuwait. Combined these represent over 3.0 million t/a of sulphur at capacity. Covid-19 is influencing the short term outlook for supply and delaying project ramp up.
- **Outlook:** DAP prices are forecast to remain firm in the coming weeks with a tight balance expected, supporting sulphur demand and pricing. Pressure is likely in subsequent quarters but the tight sulphur supply side issues are likely to prevail.

SULPHURIC ACID

- The copper price run has continued through Q1, providing support to SX-EW copper mining operations. Argus expects to see copper-based acid demand rebounding in the short term, with growth forecast at 1.8 million t/a this year on 2020 levels to 19.5 million t/a.
- Excelsior Mining announced the sale of first copper cathode from the Gunnison Copper Project in Cochise County, Arizona at the end of January. Production at the project started in December 2020 and is set to continue ramping up to reach nameplate capacity rates later in 2021. Excelsior is procuring acid from the local market to cover demand. A sulphur burner may be built in the sixth year of operation.
- Industrial-based demand in India is expected to increase in 2021. Demand from the sector contracted last year as the pandemic shutdown many industrial sectors. A recovery is expected throughout 2021 and into 2022.
- **Outlook:** Acid prices are expected to be well above 2020 levels this year – in most cases more than doubling on average. Morocco is a bright spot for trade, with expectations of a strong year for imports once again. Prices will likely be less attractive in 2021 but phosphoric acid based demand is forecast to remain healthy. The macro picture is healthy with an effective vaccine roll out in hand in major regions with significant stimulus packages and Opec+ agreement compliance set to bolster 2021 GDP growth.


Science behind Technology

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Sulphur Industry News

UNITED STATES

Freezing storm cuts refinery output

Storm Uri which blanketed the southern states of the US with snow, and led to widespread power outages in Texas, has had a major impact upon US Gulf Coast and Southern-Midwest refined products supply. Most Gulf Coast refineries were shut down or forced to operate at reduced rates. IHS Markit estimated that more than 5.2 million bbl/d of Gulf Coast capacity and 730,000 bbl/d in PADD 2 was impacted by the winter weather at its height, and close to 20% of lower 48 US natural gas production was shut-in in the first half of February. A drop in crude oil production of between 2.2 and 4 million bbl/d was estimated.

Shell's 320,000 bbl/d Deer Park refinery in Texas was reported to be likely to be offline until April. Exxon was also reported to be experiencing delays in restarting its waste water treatment unit and SRU at its 560,000 bbl/d Baytown refinery.

Private equity consortium to buy DuPont Clean Technologies

An international private equity consortium including BroadPeak Global LP, Asia Green Fund, and The Saudi Arabian Industrial Investments Company ('Dussur') has signed a definitive agreement to purchase the Clean Technologies business of DuPont de Nemours, Inc. for $510 million. As part of the transaction, Tensile Capital Management LP is providing preferred equity financing. The transaction is expected to close in the second quarter of 2021 subject to customary closing conditions and regulatory approvals.

Since first producing sulphuric acid catalyst in 1925, DuPont Clean Technologies has become a global leader in catalyst and process technologies to produce and regenerate sulphuric acid, hydroprocessing technology to desulphurise motor fuels, alkylation technology to produce clean gasoline and air pollution control systems for refineries and chemical facilities.

Nadim Qureshi, co-founder and managing partner of BroadPeak Global, said, "We are excited to have this mission-critical business in our portfolio. We intend to build on this strong foundation and further expand the business with the support of management and our strategic partners."

Eli Ben-Shoshan, Global Business Director of DuPont Clean Technologies, and future CEO of the intended independent business, said: "I'm extremely proud of the global capabilities and deep expertise of the DuPont Clean Technologies team. We believe this transaction will further strengthen our extensive global relationships to best serve our customers around the world, and I look forward to working with the consortium members to accelerate our growth."

AUSTRALIA

SRU issues at Orbost gas plant

Australia's APA Group has told investors that it is likely to take a pre-tax non-cash impairment of around A$249 million (US$194 million) due to issues experienced with the commissioning of its Orbost gas plant in the state of Victoria. APA said the impairment reflects increased capital expenditure and reassessment of the plant's future cash flows following commissioning work last year.

Orbost handles production from the Cooper Energy-operated Sole gas field, however, performance of the plant has been impaired by foaming in the sulphur recovery unit's two absorbers. This saw APA undertake works in November and December to reconfigure the absorbers to enable their operation either independently, in parallel or in series.

"The impairment charge reflects the continuation of production levels and expenditure based on the current performance of the asset since re-configuration and resumption of processing at the processing plant," APA said. APA bought the mothballed Orbost plant from Cooper Energy in 2017, with the former carrying out upgrades to the plant. However, the plant is yet to achieve peak production.

INDONESIA

Shell to license technology for new refinery

PT Pertamina Rosneft Pengolahan dan Petrokimia (PT PRPP) has signed a licensing and basic engineering agreement to apply Shell Catalysts and Technologies processes at a new refinery in Tuban, Java. The new grassroot refinery project will have a crude processing capacity of 300,000 bbl/d according to Shell, as well as a petrochemicals complex designed to produce more than 1.0 million t/a of ethylene, 1.3 million t.a of aromatic hydrocarbons, and 650,000 t/a of polyethylene. PT PRPP will use Shell's distillate hydrotreating process for the catalytic removal of sulphur from diesel fractions of crude oil.

"We are grateful that PT PRPP has selected Shell Catalysts and Technologies for their major grassroots refinery and petrochemical complex, which is a testament to the technology offerings of Shell Catalysts and Technologies and our owner-operator experience enabling PT PRPP to achieve a high return on investment," said Gurminder Singh, technology licensing director with Shell Catalysts and Technologies. The refinery is expected to be completed in 2025.

ITALY

ISAB to revamp hydrotreater

DuPont Clean Technologies has signed a contract with ISAB Srl to revamp the refinery's existing trickle bed diesel hydrotreater at the Priolo Refinery in Siracusa, using *IsoTherming®* hydroprocessing technology. ISAB commissioned the project to increase unit capacity to a target of 205 m3/hr (31,000 bbl/d), while also extending catalyst cycle length.

IsoTherming® uses a novel liquid-phase reactor system which DuPont says offers advantages over conventional hydroprocessing technologies in that it uses hydrogen and catalyst more efficiently, enabling refiners to produce low sulphur fuels that comply with increasingly stringent environmental requirements. It can cope with a wide range of feedstocks, from kerosene to vacuum gas oil, including 100% light cycle oil. Utility savings of 30-60% and capital cost savings of up to 30% can be achieved compared to trickle bed technologies.

The hydrotreater is due to start-up by 2024.

LITHUANIA

Alkylation unit for Mažeikiai refinery

DuPont Clean Technologies will supply ORLEN Lietuva in Lithuania with licensing, engineering, and technical services for a *STRATCO®* alkylation unit and *MECS®* spent acid regeneration unit at the Mažeikiai refinery. The refinery processes an average of 8 million t/a of crude with a capacity of up to 10 million t/a of crude. In order to increase

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refinery complexity and flexibility, ORLEN has commissioned an alkylation unit with 240,000 t/a (6,000 bbl/d) alkylate capacity. The alkylation unit will use LPG in the conversion to alkylate. The 75,000 t/d spent acid regeneration unit, also under license from DuPont, will provide the refinery with a consistent supply of sulphuric acid, used as catalyst for the alkylation unit, while ensuring compliance with the region's stringent emission regulations.

AZERBAIJAN

Tecnimont wins SOCAR EPC contracts

Maire Tecnimont subsidiaries Tecnimont and KT-Kinetics Technology have been awarded two engineering, procurement and construction contracts by the State Oil Company of Azerbaijan (SOCAR) for the modernisation and reconstruction of the Heydar Aliyev Oil Refinery at Baku. The overall contracts' value equals to approximately $160 million. The first contract covers the installation of an FCC gasoline hydrotreating unit, while the other is for to the installation of an LPG (mercaptan oxidation unit and an amine treatment and LPG pre-treatment unit. These units are crucial for upgrading the quality of gasoline produced by the refinery to meet a Euro-V standard. The projects are expected to be completed respectively within 33 months and 26 months from the signing date.

Pierroberto Folgiero, Maire Tecnimont Group CEO, commented: "We are really honoured to once again put our technological expertise at the service of an historical and prestigious client such as SOCAR, thus consolidating our successful, long-time collaboration aimed at unlocking greater value in Azerbaijan's downstream value chain. This achievement lets us foster a mutually beneficial relationship and paves the way to future growth, confirming the reliability of our technology-driven strategy."

SOUTH AFRICA

Investigation into alleged sulphur release

Sasol's complex at Secunda has been blamed for a 'sulphur smell' that stretched as far as Johannesburg, 60 km away. South Africa's Department of Environment, Forestry and Fisheries said that its preliminary investigations showed elevated levels of sulphur dioxide and hydrogen sulphide in the air, and said they were "most likely" to have emanated from Secunda.

In a statement, Sasol said that operations at Secunda "have been stable with no operational incidents that have/could have resulted in an increase in sulphur emissions. High resolution analysers (on point sources), which monitor our emissions, report levels below the limits prescribed by our atmospheric emissions licenses under which we operate."

It added that it was "committed to improving air quality in the areas within which we operate and has initiated an investigation to assist in identifying the area of origin of the sulphur odour experienced in the Highveld region. To this end, we are gathering and assessing data on atmospheric conditions in the region over the last few days. An initial review of data from the South African Weather Service's ambient air quality monitoring stations, as well as those managed by Sasol, indicate no exceedance of sulphur ambient air quality standard limits as set by government."

INDIA

Prime Minister dedicates new oil and gas projects

Indian Prime Minister Narendra Modi dedicated the Ramanathapuram-Thoothukudi natural gas pipeline and associated gasoline desulphurisation unit at the Chennai Petroleum Corp. Ltd (CPCL) Manali refinery on February 17th. He also laid a foundation stone for the new Cauvery basin refinery at Nagapattinam. The 143km Ramanathapuram-Thoothukudi section of the Ennore-Thiruvallur-Bengaluru-Puducherry-Nagapattinam-Madurai-Tuticorin gas pipeline is now mechanically complete. Once operational it will take gas from ONGC gas fields and deliver it gas as feedstock to the Southern Petrochemical Industries Corp. Ltd. (SPIC) site at Tuticorin as well as other industrial/commercial customers in the region. The gasoline desulphurisation unit at CPCL will produce low (<8 ppm) sulphur gasoline. The 9 million t/a refinery at Nagapattinam, meanwhile, a joint venture between the Indian Oil Company (IOCL) and CPCL, will produce gasoline and diesel to Bharat-VI specifications, as well as polypropylene as a value-added product.

The dedications were part of a slew of oil and gas project inaugurations that Modi has lent his presence to over the past couple of months, including a second 270,000 t/a catalytic dewaxing unit at IOCL's Haldia refinery, and the 348-km Dobhi-Durgapur gas pipeline, part of the Pradhan Mantri Urja Ganga project to feed the HURL Sindri urea plant, and an LPG import terminal at Haldia built by the Bharat Petroleum Corporation.

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PHOTO: IOC

IOC's refinery at Gujarat

IOC looking to sell hydrogen and sulphur recovery facilities

The Indian Oil Company reportedly plans to monetise its hydrogen-producing facilities to raise about $1.37 billion, according to local press reports. The state-owned giant is the largest refiner in India and operates one third of India's refining capacity. It is also India's largest producer of hydrogen producer. It now aims to hive off some of its hydrogen plants and sulphur recovery facilities at its refineries as a separate company which will then be part-sold to private investors.

The Indian government is looking to sell stakes in various on-core assets of state-owned oil and gas companies, and has proposed selling shares in pipelines owned by IOC, the Gas Association of India Ltd (GAIL) and Hindustan Petroleum, raising $2.3 billion. Around half of this would come from sale of IOC assets. It is suggested that IOC's offer to sell hydrogen and SRU capacity may be an attempt to deflect any sale of its pipeline capacity, which the company wishes to avoid.

Axens selected for Numaligarh expansion project

Numaligarh Refinery Ltd (NRL), a subsidiary of state-owned Bharat Petroleum Corporation Ltd, has selected Axens to supply technologies for its Numaligarh Refinery expansion project (NREP). The refinery, in the Golaghat district of Assam, was commissioned in 2000 with a capacity of 3 million t/ar to process local crude. NRL is now planning to expand refinery capacity from 3 million t/a to 9 million t/a by building a new 6 million t/a refinery with downstream grassroots facilities at the same location.

Axens has been selected by NRL to license a naphtha hydrotreating unit, a continuous catalytic reforming unit, a C5-C6 isomerisation unit, and a FCC gasoline selective desulphurisation (*Prime-G+*) unit. The scope of work includes supply of the basic engineering design package, catalysts and adsorbents, proprietary equipment, training and technical services.

Axens' Process Licensing executive vice president Patrick Sarrazin said: "The expansion of the Numaligarh Refinery will help NRL to meet growing domestic demand for clean fuels reaching BS VI specifications. We are delighted to support NRL by supplying our extensive refining technology expertise, global capabilities in basic engineering design, catalysts, equipment and services."

COLOMBIA

Ecopetrol plans Barrancabermeja refinery revamp

State-owned Ecopetrol says that it will invest $777 million over the next two years to upgrade and modernise its Barrancabermeja refinery. The works will be aimed at improving water conservation, reducing emissions and raising the quality of fuels, according to an Ecopetrol statement. Initiatives include an upgrade of the refinery's wastewater treatment plant, the refurbishment of water segregation collectors and a sulphur emission control project. The company also aims to update and expand the facility's hydrocracking unit, allowing it to reduce sulphur content in gasoline to 30 ppm in 2025 and 10 ppm in 2030. Barrancabermeja is Colombia's largest oil refinery, with a capacity of 225,000 bbl/d.

BELARUS

Hydrocracking plant commissioning

Mozyr Oil Refinery says that it is in the process of commissioning its new heavy oil hydrocracking complex, although cold weather has restricted pipeline testing. The heavy oil hydrocracking complex is the company's largest ever investment project, with a cost put at $1.4 billion. It comprises an integrated hydrocracking unit, a hydrogen plant, a sulphur recovery unit, and 21 off-plant facilities.

MALAYSIA

Sour gas project moving ahead

Petronas is moving ahead with its large Kasawari sour gas field development offshore of Bintulu, Sarawak. The company says that it is performing pre-installation surveys for the transportation and installation of the 8,600 tonne Kasawari wellhead platform which is due to be in place by the end of March. Dutch marine company Heerema Marine Contractors last year won a substantial sub-contract for the transportation and installation of the Kasawari offshore structures – the central processing platform, wellhead platform, flare structure and two bridges. Heerema will execute the transportation and installation contract in two phases, starting with the wellhead platform later this year and the other structures in 2022. Petronas is is targeting production of 900 million cfd of gas at peak.

MEXICO

New gas sweetening plant

Malaysian-based Coastal Contracts Bhd together with its Mexican joint venture partner Grupo Empresarial Alfair SAPI, says it has secured an EPC contract to build a $45 million gas processing plant in Mexico. The onshore gas sweetening plant will be constructed at the Ixachi field in Veracruz, for Petroleos Mexicanos (Pemex), and will have a processing capacity of up to 180 million scf/d of wet sour gas. The Ixachi field has reserves of 1.3 billion barrels of oil equivalent, and is the largest onshore discovery in Mexico in the past 25 years.

UNITED ARAB EMIRATES

Bids submitted for Hail and Ghasha development

EPC contractors have submitted commercial bids to the Abu Dhabi National Oil Company (ADNOC) for the four main EPC packages of the estimated $15 billion Hail and Ghasha offshore sour gas field development projects. According to the original Hail and Ghasha project plan, the four main EPC packages, their estimated contract values and the contracting entities bidding for each, are:

Package 1 (offshore) – circa $3 billion: Saipem/UAE National Petroleum Construction Co; McDermott/Tecnicas Reunidas; Archirodon/Sinopec Engineering; Petrofac.

Package 2 (offshore) – circa $2 billion: Petrofac/Samsung Engineering; Saipem/China Petroleum Engineering & Construction/NPCC; Hyundai Engineering & Construction; Archirodon/Sinopec Engineering.

Package 3 (onshore) – circa $1-2 billion: Samsung Engineering/Petrofac; Archirodon/Sinopec Engineering/Consolidated Contractors Company; Dodsal; Tecnimont.

Package 4 (onshore) – circa $4-6 billion: Petrofac/Samsung Engineering; Tecnicas Reunidas/McDermott; Tecnimont/Larsen & Toubro Hydrocarbon Engineering; Saipem/CPECC.

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Sulphuric Acid News

INDONESIA

Copper smelter deal nearing closure

Copper major Freeport-McMoRan is reportedly on verge of finalising a $2.8 billion deal with China's Tsingshan to build a copper smelter in Weda Bay, Indonesia. The Indonesian government said that the companies were aiming to conclude negotiations by the end of March. It also said that most of the financing will be borne by Tsingshan, with Freeport possibly only needing to provide funding for 7.5% of the total project cost. The smelter would have a copper concentrate input capacity of 2.4 million t/a.

In June last year, Indonesia's minister of maritime affairs and investment said the government had requested that Freeport construct a copper smelter in Weda Bay located in North Maluku in eastern Indonesia, as it seeks to build an integrated smelting hub there. There is no timescale for the completion of the project as yet. ■

Antam looking to 2024 start-up for HPAL

The Indonesian government says that it is aiming for state-owned PT Aneka Tambang Tbk to produce 50-100,000 t/a of nickel sulphate when its high-pressure acid leach plant begins operations by 2024, according to Agus Tjahjana, head of Indonesia's EV battery project acceleration team. Speaking to Reuters, he said: "nickel sulphate… precursors and cathodes… will be used primarily for the needs of domestic and export industries."

Agus also said that Indonesian production of precursors and cathodes for batteries will reach 120-240,000 t/a by 2024. The precursors and cathodes production will be spearheaded by a joint venture of PT Pertamina and Mining Industry Indonesia. Domestic electric vehicle manufacturers are aiming to start producing vehicles in 2022. Wood Mackenzie has forecast that nickel sulphate production in Indonesia could reach 450,000 t/a in 2027, driven by increasing demand for electric vehicles.

BRAZIL

Brazilian Nickel to proceed with heap leaching project

Brazilian Nickel says that it has received an installation license for PNP1000, the first phase of its Piauí nickel-cobalt laterite project in the northeast of Brazil. The permit, issued by the Piauí state environmental watchdog, allows the company to begin construction work, with first nickel and cobalt hydroxide production expected in the second quarter of 2022. The company will expand its existing demonstrator plant to produce 1,400 t/a of nickel and 35 t/a of cobalt via a heap leach process. A second phase aims to expand production to 25,000 t/a of nickel and 900 t/a of cobalt. Engineering and construction group SNC Lavalin, which produced the project's bankable feasibility study, says that ore processing at Piauí will have an "inherently low carbon dioxide footprint" compared to rival HPAL based processes. The project has been backed to the tune of $25 million by the US government,

via one of the funding partners, battery metals investment company TechMet.

EGYPT

EPC contract awarded for sulphuric acid plant

Russian project management company Nuberg EPC has been awarded an engineering, procurement and construction contract on a lump sum turnkey basis to build a 500 t/d sulphuric acid plant in Egypt. The project, is for Sprea Misr, a chemicals and plastics manufacturer in Ramadan City, Egypt. Nuberg EPC will also supply technology for the plant based on double contact double absorption (DCDA) process technology. The project also incorporates a 5MW turbine with a steam-based power generation plant with a complete bypass arrangement. Sulphuric acid produced in the plant will be supplied to the local fertilizer industry for the production of urea and other fertilizers.

This is Nuberg's fourth recent project in Egypt, including a sulphuric acid plant for Agrochem in Alexandria.

A. K. Tyagi, MD, Nuberg Engineering commented, "We are thankful to Sprea Misr for entrusting our turnkey project engineering capabilities and our EPC services and solutions with its sulphuric acid plant. We are excited to partner with them and strengthen our relationship even further. We are committed to commission the project by the first quarter of 2022, faster than the industry average of 16 months."

INDIA

Rama begins production from acid plant

Rama Phosphates Ltd says that it has begun commercial production of sulphuric acid and its derivatives from its new manufacturing facility (Unit-2) at its existing factory premises at Indore. The plant produces 55,000 t/a of sulphuric acid, as well as 9,000 t/a of chlorosulphonic acid, 20,000 t/a of liquid SO_3, 33,000 t/a of 23-27% oleum and 25,000 t/a of 65% oleum. The new facility takes the company's total acid capacity at Indore to 155,000 t/a.

Minister inaugurates iron sulphate and sulphuric acid plants

The Chief Minister of Kerala state Pinarayi Vijayan has officially opened a new iron sulphate recovery plant and a revamped sulphuric acid plant at Travancore Titanium Products Ltd (TTP) in Kochuveli, at a ceremony also attended by Industries Minister E.P. Jayarajan, TTP chairman A.A. Rasheed and managing director George Ninan.

The iron sulphate recovery plant is capable of producing 165 t/d of iron sulphate, or copperas, which is produced as a by-product of titanium dioxide manufacture at TTP. "The recovery of copperas will help TTP to come out with iron sulphate as a value-added product. Additionally, the quality of titanium dioxide produced at TTP too will improve," TTP managing director Georgee Ninan said.

The plant was constructed at a cost of $4.6 million, including $2.1 million sanctioned by the present LDF government. The copperas recovery plant would also help TTP cut down pollution, company officials said. The sulphuric acid plant, originally built in 1995, has been revamped at a cost of $1.35 million.

SWEDEN

Metso Outotec restructures Swedish operations

Metso Outotec has announced that it will cut approximately 80 positions, mainly in its Minerals, Metals, and Services business areas in Sweden. The company says that most of the layoffs will take place at its operations in the northern municipality of Skellefteå and the central municipality of Sala. Outotec is in negotiations with local trade unions concerning the restructuring and operational reduction of its operations in Sweden, relating to new product strategies and business model changes and synergy targets from the 2020 Metso Outotec merger. Outotec Oyj and Metso Corporation's Minerals business merged in early 2020, with Metso shareholders owning 78% and Outotec shareholders 22% of the combined company.

AUSTRALIA

Potassium sulphate output to begin in April

Australia's first potassium sulphate (also known as sulphate of potash or SoP) manufacturer SO4 says that it expects to begin production in April at its site at Lake Way, Western Australia. Initial output will be a water soluble premium grade of SoP, followed by a granular grade in September. Together with new production from Kalium Lakes Ltd (KLL) at Beyondie, 180 km further north, currently 80% complete, the two companies are expected be able to supply all of Australia's current and expected SoP needs for at least the next 30 years. Both SO4 and KLL will make SoP fertiliser from potassium-rich brine abstracted from beneath the salt crusts on Lake Way and the Ten Mile and Sunshine lakes at Beyondie. SO_4 has binding offtake agreements with fertiliser industry partners for 220,000 t/a, or around 85% of its initial planned production. KLL expects to start production in September and aims to initially produce 90,000 t/a of SoP fertiliser before ramping up to 180,000 t/a. Demand for SoP produced by solar evaporation of salt lake brine rather than the conversion of muriate of potash (potassium chloride) via reaction with sulphuric acid at high temperature, is expected to increase as countries move closer to 2050 and 2060 environmental targets.

MADAGASCAR

Ambatovy looking to March restart

Sumitomo Corp has confirmed that it plans to resume operation at its Ambatovy nickel plant in Madagascar in March as planned, aiming to produce 3,000 tonnes of nickel during Q1. Sumitomo may book an additional impairment loss on the project depending on the metal price and its output plan which is being reviewed, according to Hiroyuki Sugai, assistant CFO of the company.

BULGARIA

Decrease in Pirdop smelter output

Aurubis said that the copper concentrate throughput of its smelter in Pirdop, 80 kilometres east of the capital Sofia, decreased by 2% to 327,000 tonnes in the first quarter of its fiscal year 2020/2021, which started on October 1st. Cathode output at Pirdop rose 4% year-on-year to 57,000 tonnes. in the October-December period of 2020, Aurubis said in an interim financial statement published last week. Sulphuric acid output at Pirdop was 332,000 tonnes for the quarter, down 1% on the year.

JORDAN

Indo-Jordan Chemicals boosts phosphoric acid production

A new $6 million expansion project has begun operation at the Indo-Jordan Chemicals Company site at Al-Shediya in the governorate of Ma'an in southern Jordan. Indo-Jordan chemicals is a wholly-owned subsidiary of Jordan Phosphate Mines Co. (JPMC). The expansion will increase the company's output of phosphoric acid by 24,000 t/a. ■

People

During the 10th Russian Corporate Governance Forum, **Andrei Guryev**, PhosAgro's CEO and a member of the company's board, was declared the winner in the category: "Contribution to the Development of ESG Culture". PhosAgro's Corporate Secretary, **Sergey Samosyuk**, was named the winner in the category "Corporate Governance Director/Corporate Secretary".

"For PhosAgro, a global company that interacts with a wide range of international stakeholders – from investors and regulators to customers in 102 countries around the world – a commitment to ESG principles, transparency and disclosure, as well as improving corporate governance standards, are clear priorities," said Mr Rolet about the award results.

Umicore SA is looking for a new chief executive officer to lead the world's largest maker of electric-vehicle battery materials, after announcing the planned departure of veteran leader **Marc Grynberg**. The Belgian company's board has started an internal and external search and likely will announce an appointment in the coming months.

"You need to have a pretty solid constitution and a high degree of serenity in this job to not get carried away when things are going really well and not get pessimistic when things are tough," Grynberg said.

Grynberg is stepping down after presiding over an almost sevenfold increase in Umicore's valuation over more than 12 years. The incoming CEO will oversee construction of Umicore's new EV cathode plant in Poland, the first in Europe.

Arianne Phosphate has appointed **Pier-Elise Hebert-Tremblay** as the Company's CFO, effective immediately. Ms. Hebert-Tremblay will replace **Andrew Malashewsky** who had held the position since January 2019. Ms Hebert-Tremblay has been a member of the Certified Professional Accountants of Quebec and Canada since August 2011 and holds a bachelor's degree in accounting and a Master's in Business Administration from the University of Quebec at Chicoutimi. She served as Financial Controller of Arianne Phosphate since 2010 and specialised in financial reporting compliance for junior listed resource companies, forecast development and financial modelling.

"Pier-Elise has been involved with Arianne almost since its inception and advanced her way through the Company, proving herself to be a very valuable member of the team," said Brian Ostroff, CEO of Arianne Phosphate. "I have had the opportunity to work closely with Pier-Elise since my time as CEO and, as CFO, I have no doubt she will play an integral part in advancing Arianne's efforts to secure financing and partners as it moves its Lac à Paul project towards development. With the recent significant upturn in the agricultural sector and increased interest in Arianne, the timing couldn't be better."

Itafos has announced the appointment of **David Brush** as Chief Strategy Officer. Brush has been serving as a consultant since December 1st, 2020. In his role as Chief Strategy Officer, he will be responsible for strategy development and implementation, including related capital structuring and corporate development initiatives.

Brush has over 30 years' experience in all aspects of global business operations, business development and strategic planning, financial management, personnel development, and general management. Prior to joining Itafos, he was founder and Managing Partner of Idris Capital where he advised clients on M&A transactions and restructurings across a variety of sectors, including agriculture, fertilizer, packaging, building products and consumer products.

Berndorf Belt Technology USA (BBT) has named **Jeff Dallstream** Sales Executive of the Western Region North America. He supports their position as leader in steel belt production, process equipment technologies, and customer support. In his new role, Dallstream is responsible for supporting the sales of process equipment for SBS Steel Belt Systems USA (SBS USA), in his region, recently acquired by Berndorf Band Group. Dallstream is an industry veteran, with over 15 years' experience. After college he entered the steel belt and steel belt processing equipment industry as a design engineer for North American Process Systems which became Berndorf Steel Belt Systems. Dallstream has worked with multiple companies in sales support or as a regional sales manager selling carbon steel and stainless-steel belts and the associated process conveying systems for the chemical, food and fertilizer industries. Dallstream also has 12 years running a division of an equipment fiancé company in Scottdale, AZ arranging the financing of fertilizer based stainless steel belt systems among many other types of equipment ranging from aircraft to yellow iron. ■

Calendar 2021

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

MARCH

23-25

Phosphates 2021 Conference – **virtual event**
Contact: CRU Events,
Chancery House,
53-64 Chancery Lane,
London WC2A 1QS, UK.
Tel: +44 (0) 20 7903 2444
Email: conferences@crugroup.com

APRIL

19-23

Brimstone Amine Treating and Sour Water Stripping – **online training course**
Contact: Mike Anderson, Brimstone STS
Phone: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

MAY

3-7

Brimstone SRU Maintenance and Reliability – **online training course**
Contact: Mike Anderson, Brimstone STS
Phone: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

10-14

Brimstone Amine Treating Training – **online training course**
Contact: Mike Anderson, Brimstone STS
Phone: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

17-21

Brimstone Sulphur Recovery Training – **online training course**
Contact: Mike Anderson, Brimstone STS
Phone: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

19

The Sulphur Institute Sulphur World Symposium – **virtual event**
Contact: Sarah Amirie,
The Sulphur Institute
Tel: +1 202 331 9660
Email: sulphur@sulphurinstitute.org
Web: sulphurinstitute.org/symposium-2021/

JUNE

Date T.B.A.

European Sulphuric Acid Association General Assembly – **virtual event**
Contact: Francesca Ortolan, Cefic
Tel: +32 2 436 95 09
Email: for@cefic.be

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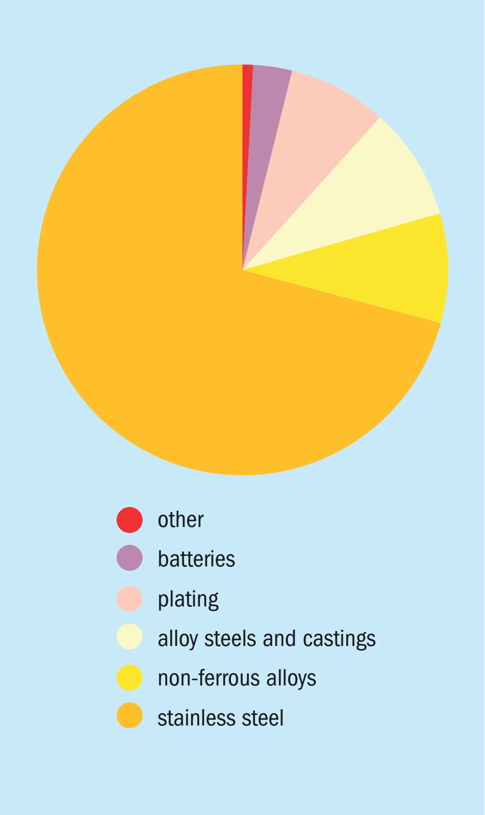
The Ambatovy HPAL plant, Madagascar.

PHOTO: AMBATOVY

The changing nickel market

Demand for nickel sulphate is expected to rise rapidly this decade to feed increased battery production for electric vehicles. At the same time, diminishing sources of high grade nickel ores are leading to a renewed focus on high pressure acid leaching (HPAL) of laterite ores, with a significant impact on sulphur and sulphuric acid demand.

Fig. 1: **End uses for nickel, 2019**



Source: Nickel Institute

Nickel sulphate has become something of a gold rush for mining companies, with a plethora of new projects announced and under development. In turn this promises to increase sulphuric acid demand for leaching of nickel ores, with knock-on effects on sulphur demand over the next decade. But will the market hype translate into real increases in consumption?

At present, according to the International Nickel Study Group, the world market for nickel reached 2.3 million t/a in 2019. Of that, around 70% of all nickel produced is used as an alloying component for stainless steel production, another 16% is used in other alloys and castings, and a further 8% for nickel plating of objects (see Figure 1). Only 4% (92,000 t/a) was used in battery production, but that figure is rising rapidly as the market for electric vehicles increases. CRU forecasts that total nickel demand could reach 2.8 million t/a by 2023, by which time nickel demand for batteries would have grown to 240,000 t/a, while the Bank of America puts nickel demand for batteries at 690,000 t/a by 2025. In its Nickel Sulphate Market Outlook to 2028 report, Roskill estimated nickel sulphate production in 2018 to have risen as much as 21% to nearly 160,000 t/a, up from less than 50,000 t/a in 2010, or 3% of the total nickel market.

The rise of EVs

Electric vehicles have been around for many years. In the UK, lead-acid battery powered vehicles for delivering milk (‘milk floats’) used to be a common sight on suburban streets, and in 1967 the UK Electric Vehicle Association boasted that the country had more electrically powered vehicles than the rest of the world put together. However, outside of such fleet vehicles operating over short distances from a central depot, the uptake of EVs was hampered by the low power density and range of contemporary batteries. The development of lithium ion batteries in the 1990s, coupled with increasing oil and gasoline prices and increasing concerns about the environment spurred a second wave of EV designs, hampered in the US by arguments over California’s attempt to create a zero emission vehicles mandate. For a while the pendulum swung towards hybrid gasoline/electric vehicles, typified by the success of the Toyota Prius, launched in 1997. This took off particularly in Japan, where 20% of all passenger vehicles are now hybrids.

But more recently, international government plans to decarbonise road transport

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and continuing improvements in battery technology are leading to a resurgence in electric vehicle use. China has been a particular pioneer, as it tries to improve air quality in its smog-ridden cities, but legislators have looked towards removing fossil fuel powered vehicles from the road altogether, with most countries committed to phasing out sales of new fossil fuel powered vehicles completely by 2030-2040, and many major car manufacturers committing to be all-electric by 2030.

Electric vehicle sales were around 130,000 units in 2012. By 2019 this had risen to 2.2 million, and in spite of the coronavirus pandemic, 2020 saw a huge leap in sales of 43% to surpass 3 million units, in spite of overall car sales slumping by 20% across the year as people drove less. China led the way, with sales of 1.3 million vehicles, followed by Germany (0.4 million), the US (0.3 million), and France and the UK (0.2 million each).

Deloitte forecasts that by 2030, battery electric vehicles will be outstripping hybrid sales by 4:1, and are likely to reach 23 million units globally, about half of which will be sold in China, 27% in Europe and 14% in the United States (see Figure 2). This represents an average annual growth rate of 29%.

Changing battery chemistry

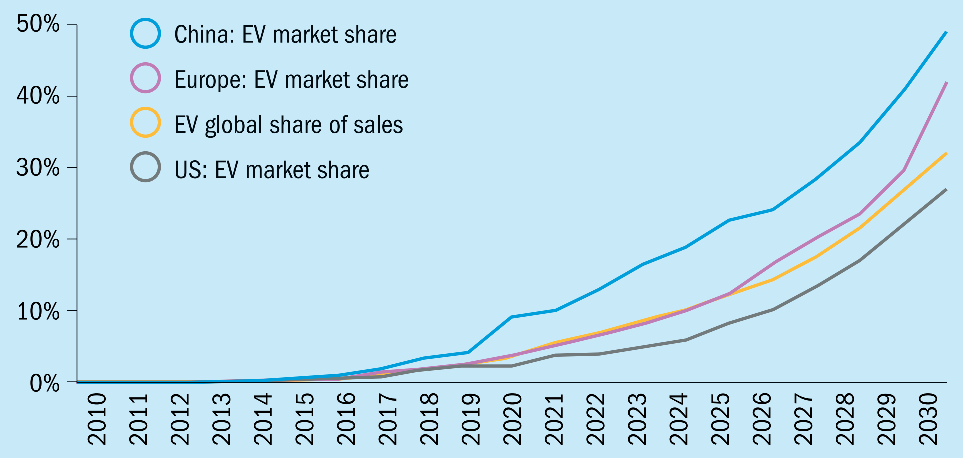
At the same time that electric vehicle use is taking off, nickel requirements for batteries are also changing. Two chemistries of lithium-ion batteries dominate electric vehicle batteries: nickel-manganese-cobalt ('NMC'), and nickel-cobalt-aluminium ('NCA'). NMC batteries are used by most vehicle manufacturers, the main exception being Tesla, which uses NCA batteries. NCA batteries already have a high nickel content – the ratio of nickel to the other metals in an NCA battery is around 80%. NMC batteries began at around 33%, but are also steadily moving towards more nickel-rich formulations, from the original nickel-manganese-cobalt ratio of 1-1-1 to a 6-2-2 and even 8-1-1 (80%) ratio, the latter comparable with NCA batteries. The reason is that nickel offers greater energy density and storage at lower cost, delivering a longer range for vehicles, currently one of the restraints to EV uptake.

Class 1 nickel

Lithium-ion batteries with nickel-rich cathodes require high purity nickel, typically in the form of nickel sulphate. As the main feedstock for EVs battery cathodes, nickel sulphate is currently manufactured by dissolving so-called 'Class 1' nickel (nickel with greater than 99.8% metal purity) in sulphuric acid. However, nickel cathode production is steadily gravitating towards bypassing the metallic stage of Class 1 nickel and using purified nickel sulphate in solution generated during the nickel refining process.

Nickel deposits around the world exist in two main forms; nickel sulphide deposits, and nickel laterite deposits. The latter is an oxidised form, generally found in tropical climes north and south of the equator, including the Caribbean, and South America, southeast Asia and western Pacific, and central Africa. It is further subdivided into limonite and saprolite ores, depending on the relative levels of magnesia, silica and iron content. Laterites are however generally lower grade in terms of nickel (<1% Ni) than sulphide deposits. Up until now, laterites, especially iron-rich laterites, have mainly been used for production of lower grade nickel for stainless steel production. In particular this led to the production by China and Indonesia of so-called 'nickel pig iron' (NPI) – a ferronickel agglomeration which can be used directly in stainless steel manufacture. A pyrometallurgical process could also be used to produce similar but slightly higher grade ferronickel.

Fig. 2: **Electric vehicle share of new car sales, 2010-2030**



Source: Deloitte

Meanwhile, Class 1 nickel used to be made predominantly from high grade nickel sulphide deposits. However, sulphide deposits are rarer, representing only around 19% of all nickel deposits, and many have already been mined out. At present only around 29% of all nickel production comes from sulphide deposits (although 60% of Class 1 nickel production). There are some new sulphide deposits, such as are aimed to be exploited by the Turnagain Project in British Columbia, and the Dumont Project in Québec. However, these are insufficient to meet the increasing requirements for Class 1 nickel going forward. Some forecasts put Class 1 nickel demand for EVs at 2.6 million t/a by 2040, more than all nickel production combined today. In its baseline outlook, Roskill forecast nickel sulphate demand to continue to grow to more than 900,000 tonnes Ni by 2029, boosted "not merely by rising adoption rates of EVs, but also by increased vehicle range (and battery size), and the increased intensity of use of nickel among various competing battery chemistries, growth in nickel sulphate demand is set to outpace that of most other battery raw materials". Wood Mackenzie were slightly more cautious, putting nickel sulphate demand at 800,000 t/a (Ni basis) by 2035.

Elon Musk, owner of the Tesla car company (amongst his many projects), laid this out in stark terms at his so-called Battery Day event in September 2020. He said that he was aiming to be producing 20 million vehicles per year by 2030, and that in order to do so, his main raw material supply concerns are lithium (relatively abundant) and nickel. Each Tesla model uses an average of 45 kg of nickel metal. Musk's planned production volume would thus represent 900,000 t/a of Class 1 nickel. Musk ended with a plea to nickel

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Table 1: **HPAL projects**

Operator	Capacity (t/a Ni)	Location	Start-up date	Notes
Sherritt/GNC	49,000	Moa Bay, Cuba	1959	
Norilsk	9,000	Cawse, WA	1998-2006	Closed
Glencore	45,000	Murrin Murrin, WA	1999	
Preston Resources	10,000	Bulong, WA	1999-2003	Closed
Sumitomo	24,000	Coral Bay, Philippines	2005	Expanded 2009
BHP Billiton	36,000	Ravensthorpe, WA	2007-2017	Sold
FQM			2020	Restart
Vale	58,000	Goro, New Caledonia	2010	Sale failed
Sumitomo (48%)	60,000	Ambatovy, Madagascar	2012-20	
			2021	restart
MCC	33,000	Ramu, Papua New Guinea	2012	
Sumitomo	36,000	Taganito, Philippines	2013	
New projects				
Ningbo Lygend	37,000	Obi Island, Indonesia	2021	
Tsingshan	50,000	Morowali, Indonesia	2022	
PT Huayue	60,000	Morowali, Indonesia	2022	
Clean TeQ	20,000	New South Wales, Australia	n/a	
BASF/Eramet	42,000	Weda Bay, Indonesia	2025	Feasibility study

Source: BCInsight

miners: "Well, I'd just like to re-emphasise, any mining companies out there, please mine more nickel… Tesla will give you a giant contract for a long period of time, if you mine nickel efficiently and in an environmentally sensitive way. So hopefully this message goes out to all mining companies. Please get nickel."

HPAL

With sulphide deposits limited, ways will have to be found of processing laterite ores to produce Class 1 nickel. Purification of Class 2 nickel produced via NPI or ferronickel is prohibitively expensive at present. This leaves only one remaining way to produce Class 1 nickel from laterites – the use of high pressure acid leaching (HPAL) of iron rich (limonite) ore. Heap leaching or chloride leaching have also been investigated, but heap leaching requires a very low iron content, and leaching at atmospheric pressures tends to require much more sulphuric acid than HPAL, to the extent that costs become comparable or potentially higher.

Leaching of metal from copper oxide is an established technique, via the solvent extraction/electrowinning (SX/EW) process. However, nickel is more tightly chemically bound than copper, and consequently requires far more intensive conditions to extract it via acid leaching. As a result, HPAL is forced to use sulphuric acid at >240C and 33-35 atmospheres pressure in titanium autoclaves. The intensely corrosive conditions make for challenging operation, and HPAL is widely known as a technically demanding and difficult process. It is also expensive compared to other methods of nickel extraction, which limited its application when the first major wave of HPAL projects started up in the 1990s.

Table 1 shows previous and planned HPAL projects worldwide. While there have been some notably successful operations, including Sherritt at Moa Bay, Cuba and Sumitomo at Coral Bay and Taganito, Philippines, both of which have been expanded during their time of operation, there have also been some notable failures, such as Cawse and Bulong in Australia, and many of the other operations have struggled to reach nameplate capacity and often lost money. Cawse passed from Centaur to OM Group to Norilsk before finally being axed. Vale has suffered persistent operational problems at its Goro plant on New Caledonia, culminating in a major acid spill in 2014 which has seen local opposition to the plant rise. The Brazilian major has twice attempted to dispose of its Goro site, first to New Century Resources and more recently to a consortium led by Trafigura, but both sales fell through last year, and Vale has frequently said that it will close the site down. Ramu has been plagued by accusations of waste dumping and battles with environmental regulators, and Ravensthorpe was shut down for several years until brought back online by new owners First Quantum last year.

Beyond the technical difficulties, one of the issues with laterite processing via HPAL is 'ESG', short for environmental, social and corporate governance. HPAL generates an acidic slurry which must be neutralised and properly dealt with. The liquid effluent from an HPAL operation far exceeds any facility's ability to recycle, so large quantities of solution must be disposed of, via tailings ponds or ocean discharge of liquids, depending on access to sites for so called deep sea tailings placement (DSTP), which has been contrpversial. Some facilities such as Ramu Nickel discharge the tailings at the same pH as the ocean, making them effectively inert, but others discharge acidic waste with a knock-on effect on sea life. Projects without access to deep sea disposal also face potential issues with liquid residues.

Western Australia has an advantage as the dry conditions allow for net evaporation of the solution and safe stockpiling of the dry residue. Countries with significant rainfall such as Indonesia and no deep water access must build complex and significant on-land storage facilities and ensure that liquid effluent is properly treated before return to any natural waterways. This can add $500 million to the capital cost and $0.50-1.00/lb (ca $1-2,000/t) to the operating cost.

Nickel prices

Because of the expense of new nickel production, especially via HPAL, one of the issues with the nickel market over the past few years, and the reason behind some of the HPAL shutdowns noted above, has been oversupply of nickel and low prices which have deterred new investment. However, the past year has seen nickel prices rising steadily, from a low of around $11,000/t in March 2020 to highs above $19,000/t in February 2021, in no small part due to expectations of higher demand for nickel batteries (battery sales increased nickel demand by 5% last year), as well as a generally improving macroeconomic forecast as Covid vaccines offer the prospect of a return to something like ‘normal life’ once more. The industry operates a rule of thumb that a nickel price of $20,000/t is sufficient to justify investment in new supply, and the market is certainly approaching that, with supply remaining tight.

Moreover, because of the shortage of Class 1 nickel there are premium prices available for nickel sulphate, which in February 2021 reached record levels in China of 35,000-38,000 yuan ($5,414-5,878) per tonne, its highest level on record.

New projects

A number of new nickel sulphate plants are under construction to feed the Chinese market. Some are modifications to existing facilities. BHP is also adding what will eventually be a 100,000 t/a nickel sulphate facility at its Kwinana refinery, with the first 22,000 t/a stage due on-stream this year. Powdered refined nickel from the nickel smelter will be reacted with sulphuric acid from the Kalgoorlie smelter to produce nickel sulphate for battery use. In Finland, Terrafame is looking at converting its biological leach operation at Sotkamo from intermediates to nickel sulphate production to feed European battery demand.

But over and above this, and in spite of the aforementioned patchy record of HPAL plants, there are now a number of HPAL projects, mainly in Indonesia. Chinese mining firm Ningbo Lygend is aiming to complete its project on Indonesia’s Obi island in 1Q 2021, with a capacity of ‘MHP’ (mixed hydroxide precipitates), including 160,000 t/a of nickel sulphate (50,000 t/a nickel) and 20,000 t/a of cobalt sulphate, via an HPAL process.

Tsingshan Holding Group has an HPAL project at Morowali on the Indonesian island of Sulawesi, called PT Hua Pioneer Indonesia. The facility is designed to produce 37,000 t/a of nickel and around 4,000 t/a of cobalt as mixed hydroxide precipitates. The project has been delayed until at least 2022, and recently said that it would not use deep sea tailings placement to dispose of waste from the facility and is instead seeking “other technological options”.

Tsingshan is also partnering Chinese battery manufacturer GEM, and Brunp/CATL in the PT QMB New Energy Materials project at Morowali, with another 50,000 t/a (nickel) mixed oxide precipitate process, and Tsingshan also has a 10% stake in another HPAL project, PT Huayue, with Chinese cobalt producer Zhejiang Huayou and China Moly. This is looking to 60,000 t/a of mixed nickel hydroxide cobalt production at a cost of $1.3 billion, and also decided recently not to use deep sea tailings placement. PT QMB New Energy Materials intends to process the battery-grade nickel intermediates to nickel sulphate on site, while PT Huayue will export MHP output for conversion to nickel sulphate in China.

Sumitomo Metal Mining (SMM) said in November last year that it was on track to finish a definitive feasibility study and make a final investment decision on the Pomalaa nickel project in Indonesia by the end of March 2020. This would be a partnership with PT Vale Indonesia to build a 40,000 t/a mixed nickel sulphide HPAL plant by about 2025.

Meanwhile, in Australia, Clean TeQ is aiming to produce an average of 20,000 t/a of nickel and 4,500 t/a of cobalt (as sulphates), as well as scandium oxide and an estimated 50,000 t/a of ammonium sulphate. The $1.4 billion project will include a sulphur burning acid plant to feed the HPAL autoclaves. Clean TeQ says that the ore body has a low acid consumption compared to other HPAL projects, and at capacity should require 660,000 t/a of sulphuric acid. The project has produced a final execution plant but is still securing funding and has no start-up date as yet.

BASF and Eramet have initiated a feasibility study on building an HPAL plant at Weda Bay, Indonesia with a base metal refinery at a location to be determined. The HPAL plant would process locally secured mining ore from the Weda Bay deposit to produce a nickel and cobalt intermediate. Projected capacity is 42,000 t/a of nickel and 5,000 t/a of cobalt, with a potential start-up in the “mid-2020s”.

Acid impact

Sulphuric acid consumption in HPAL is around 260-400 kg/tonne of ore processed, depending on the rock grade and reaction conditions. The three projects in Indonesia that are currently under development thus have the potential to add a considerable additional requirement for sulphuric acid. For example, Ningbo Lygend acid requirements are projected to be approximately 1.4 million t/a at capacity, and the two projects at Morowali could each require 1.7-2.0 million t/a of acid.

However, as we have noted above, HPAL is a notoriously fickle process. Historically it has required an average of four years to achieve 80% capacity for existing HPAL producers, and although some (eg Coral Bay) have been considerably faster, Murrin Murrin in Australia took seven years to achieve more than 50% capacity. This would imply on average an extra 2.5 million t/a of acid consumption by 2025, assuming that a solution can be found for dealing with the waste from the plants now that deep sea tailings placement has been ruled out. Even this might not actually significantly increase Indonesia’s sulphur or sulphuric acid imports, however, as new copper smelter capacity is also under development over the same timeline, now that Indonesia has severely restricted exports of copper and nickel concentrate in an attempt to capture more value domestically. Much will depend upon the relative timing of the various projects.

However, looking out towards 2030, the rapidly increasing demand for Class 1 nickel as nickel sulphate may well spur the development of more new projects, of which Clean TeQ and the recently announced BASF/Eramet development may be merely the tip of the iceberg. ■

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Refining in the time of Covid

The profound demand shock caused by Covid-related lockdowns has had a major impact upon the refining industry. Run rates have been at low levels in North America and Europe, and a new wave of rationalisation is under way, at the same time that capacity continues to grow in Asia. Will this spur diversification into petrochemicals and low carbon options for Atlantic basin refiners?

Refining is moving to Asia: Hengli Petrochemical's Dalian complex, China.

The past year has been one of the most difficult in some time for the refining industry. The sudden removal of several million barrels per day of demand sent a tremendous shock through the oil and refining sectors. Demand contracted by 20% as lockdowns hit first China, then Europe and North America in early 2020, and although it has recovered since then, overall demand for 2020 was down 9 million bbl/d on 2019's figures. Covid lockdowns continue to weigh upon demand, with monthly demand for January 2021 the equivalent of 6 million bbl/d lower than January 2019.

Hardest hit have been fuel demand, especially aviation fuel, with personal travel badly affected – jet fuel demand was down 40% in 2020 compared to 2019. Gasoline demand is down 10% in the US, which represents one third of all global gasoline consumption, and the US Energy Information Adminstration (EIA) predicts that it will never return to the peak of 2018.

Supply remained robust until the drastic fall in prices of April 2020, when Brent prices reached $18/bbl, at which time there was an oversupply of around 20 million bbl/d in the market. This triggered a fall in supply of an estimated 13-14 million bbl/d in May, mainly due to cuts among OPEC and allied nations, as well as price-related shut-ins among US shale oil producers.

The cuts in production have seen a recovery in prices back to $50/bbl, and, more recently, when OPEC agreed not to reverse any of its production cuts for April 2021 (except for 150,000 bbl of exemptions for Russia and Kazakhstan), oil prices rose to $70/bbl. Nevertheless, OECD commercial inventories remain at high levels and, although there have been draw-downs in recent months, they are still 150,000 barrels above pre-Covid levels.

The 'rebound'

Although oil demand took a huge hit in the early part of the crisis, it actually rebounded fairly quickly on a month by month basis to about half of that initial shock by July to September and has been relatively flat since then. OECD demand was hit much harder than non-OECD demand, which recovered quickly, especially in China. China is the only major country in the world to see year-on-year growth in oil demand in 2020 – up by just 0.3% to 14.8 million bbl/d. Although the general consensus is that it could take another 2-3 years for oil demand to regain its pre-crisis levels, if ever, there seems to be a general agreement among forecasters that 2021 may see an overall rebound in demand of up to 5 million bbl/d.

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However, gaining control over the Covid-19 outbreak is clearly essential for demand to recover beyond that, and the signs for that have been mixed. On the one hand, global covid cases peaked in early January 2021 and have been declining since then, bringing improved market sentiment. However, there are risk factors that remain, including more infectious variants. Vaccination rollouts are a positive factor, especially in the US and UK, and with improved distribution elsewhere should hopefully begin to bring the infection under control. However, the rollout has been slower than anticipated in Europe, and there are huge populations to be inoculated in places like India. Covid measures are still in flux and in the UK, for example, where the pace of vaccination has been among the highest, the government has only just begun easing a lockdown that has lasted for almost five months, and does not foresee a full relaxation of anti-Covid measures until late July at the earliest.

The IEA said in a recent monthly market report that a recovery in demand would outstrip production in the second half of the year, prompting "a rapid stock draw" of the glut of crude that has built up since the pandemic began. At the same time, though, the IEA trimmed its forecast for global oil demand for 2021 by 200,000 bbl/d to 96.4 million barrels, around 3% less than in 2019. OPEC also reduced its own 2021 demand forecast, cutting it to 96.1 million barrels a day. In a recent oil and downstream market seminar, Argus said that global demand recovery for primary fuels will be slow as uncertainties begin to clear, and that forecasts are erring towards the conservative side.

One of the great questions over future consumption is whether new working patterns and ways of going about business will have a long term effect on consumption of, eg, gasoline or jet fuel. Products like diesel and petrochemical feedstocks have shown remarkable demand resilience during the lockdowns and some growth, but others sectors are significantly down and likely to remain so. Jet fuel in particular remains particularly sensitive, as covid must be contained at both the start and end point of an aircraft's journey for flights to return to something approaching normal. But oil demand peaked in the US and Europe in 2005 and 2006, respectively, and with declining demand in both regions, another significant round of refinery rationalization has long been forecast, and now seems to be upon us.

Impact on refineries

At the refinery level, the pandemic has accelerated the eastbound shift in refining capacity and refinery run rates that had been in progress for many years. Crude oil demand is steadily moving to Asia, and especially China. China's total refinery throughput is now rapidly approaching that of the US, and may overtake it this year. In the US, low refinery activity will rebound as pandemic control measures are lifted. However, the negative demand shock of the pandemic has accelerated the retirement of some of the country's older, smaller or less competitive refineries or their conversion into renewable fuel facilities. In addition to the 7-800,000 bbl/d of refinery capacity that has been idled or mothballed, it is estimated that another 780,000 bbl/d in the US is in the process of being converted to renewable feedstocks and alternative processing. US refinery margins have been squeezed and utilisation rates are low as a consequence – below 85%. Going forward, restrictions in drilling permits on federal land announced by the Biden administration could also impact upon the US refining margins.

Across the Atlantic operators also face difficulties. European utilisation has increased marginally since the second half of 2020, but remains close to 30-year lows. European run rates are approaching technical minimums and stocks remain high – demand needs to recover for margins to improve, unless there is rationalisation. European refiners are geared towards producing middle distillates, so the fall in jet fuel demand has been a particular issue. It is also a region already oversupplied on gasoline, and has seen a full year of historically low refining margins. Diesel margins have been below $7/bbl for the longest period since 2003, and overall margins have averaged below $5/bbl – barely above operating costs. The IMO low sulphur regulations for shipping have meant that prices for fuel oil have also been very low. Low margins have forced Europe's refiners to begin rationalisation, with nearly 1 millon bbl/d of crude distillation capacity either mothballed, permanently closed or marked for conversion to renewable fuel processing.

Elsewhere, refineries in the Philippines and Oceania have also either already announced closures or are seriously considering it, leaving them dependent on imports to meet most of their oil demand needs, and again possibly giving a boost to Chinese refiners who may be well placed to step in with their own supply.

Globally, Covid-related capacity rationalisation is underway. Around 2.5 million bbl/d has already been idled or mothballed, mostly in the US, Europe, Southeast Asia and Oceania. IHS Markit forecasts around 3.7 million bbl/d of refinery closures over the period 2020–25, at the same time that 5.5 million bbl/d of new refining capacity is simultaneously being added in other regions, mainly Asia.

Feedstocks

As product demand has shifted, so has refiners' feeds. In Asia there has been a move back towards Middle Eastern crudes, as supply from non-OPEC sources slows down and prices for Arabian oil remains attractive. This has been notably true of, for example South Korea and Japan, whose efforts to diversify their crude import sources have reversed to a reliance on Middle Eastern grades last year.

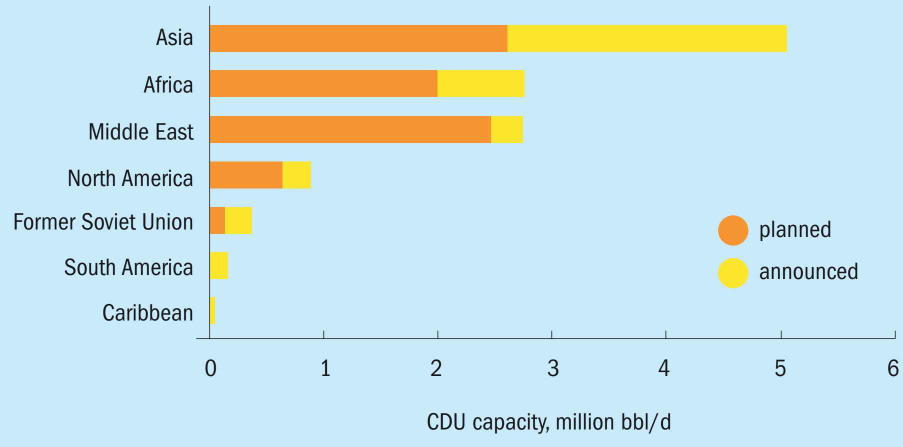
Reduction in US shale oil production has had a major impact upon the market, with production still down 1.5 million bbl/d from where it was a year ago, although, oddly, exports have not fallen to the same degree, and remain at around 3 million bbl/d, mostly to Asia and Europe. The southern Permian basin has been the most resilient of the shale regions, and forecasts are for production to return as oil prices rise. WTI prices of $60/bbl or above are judged to be sufficient to lead to an extra 500,000 bbl/d of new production each year, and could lead to US crude exports rising to 4 million bbl/d by 2023.

In the meantime, the US is importing more Canadian heavy crude, in spite of the final cancellation by the Biden administration of the Keystone XL pipeline. There are a number of other routes for Canadian crude into the US now, and expectations are for US imports of Canadian crude to rise. Some of this has even been re-exported via the US Gulf last year.

Diversification

In such a difficult economic environment for refiners, especially in North America and Europe, operators are looking at ways of changing the way that they operate, perhaps to capture value from carbon credits via hydrogen production and biofuels, or integrating higher value petrochemicals into their production. On the petrochemi-

Fig. 1: **Planned and announced refinery CDU capacity additions by region, 2020-24**



Source: GlobalData, Oil and Gas Intelligence Center

cal side, IHS Markit has suggested that for many refiners propylene, C4 streams, aromatics, and feedstocks such as liquefied petroleum gas (LPG) and naphtha may be available in sufficient quantities to develop downstream chemical production, depending upon local markets – fitting into local value chains by substituting imports or finding alternative uses for relatively low-value refining streams can lead to attractive propositions. They acknowledge however that not all refinery-integrated chemical opportunities are large, and some do push refiners to venture into an industry they are not used to. Collaboration with partners more familiar with the industry, such as traders to secure product offtake, or local chemical producers who could help mitigate the perceived risk of integrating into chemical production, can help ease the transition.

However, there are a number of other options for refiners to move beyond their traditional activity, including energy transition projects such as carbon capture, utilisation, and storage (CCUS); hydrogen production; desalination; gas import and supply; plastics recycling; traditional power projects; and renewable power, including waste-to-energy.

Decarbonisation

One of the pressures that refiners are having to face is attempts by governments, particularly in Europe, to reduce carbon emissions from transport and industrial sectors. The EU's Renewable Energy Directive (RED II) originally assumed a 14% reduction in emissions from transport by 2030, but it is looking likely that this will become more ambitious and may be 20% or higher. In the US, the Biden administration is also looking to speed up moves on tackling carbon emissions, with the possibility of introducing a carbon pricing scheme. Longer term, there are incentives to look elsewhere than gasoline and diesel. The European car fleet is expected to be 20% composed of electric vehicles by 2030 and 50% by 2040. Bans on sales of new internal combustion engined vehicles are expected in Norway in 2025, Denmark, Sweden, Slovenia, Netherlands and Ireland by 2030, the UK by 2035 and France and Spain by 2040.

However, while there are legislative pressures towards refiners changing towards energy transition pathways, there are also economic incentives. For example, Total said its biorefinery at La Mède, which began production in 2019, was the only one of its European units to turn a profit in 2020.

Decarbonising refineries is not an easy step. Some progress can potentially be made via energy efficiency schemes, which not only might secure carbon credits but also bring cost savings from reduced energy consumption. Refineries offer a number of processes that require significant amounts of heat, and recovering and utilising heat that would otherwise be wasted can be a sound investment, e.g., via upgrading an inefficient boiler system. Renewable electricity can also be used to replace natural gas or coal-fired heating. In theory, up to 70% of processes that currently burn fuel in refineries could be replaced with electricity, albeit at a capital cost of replacing the equipment.

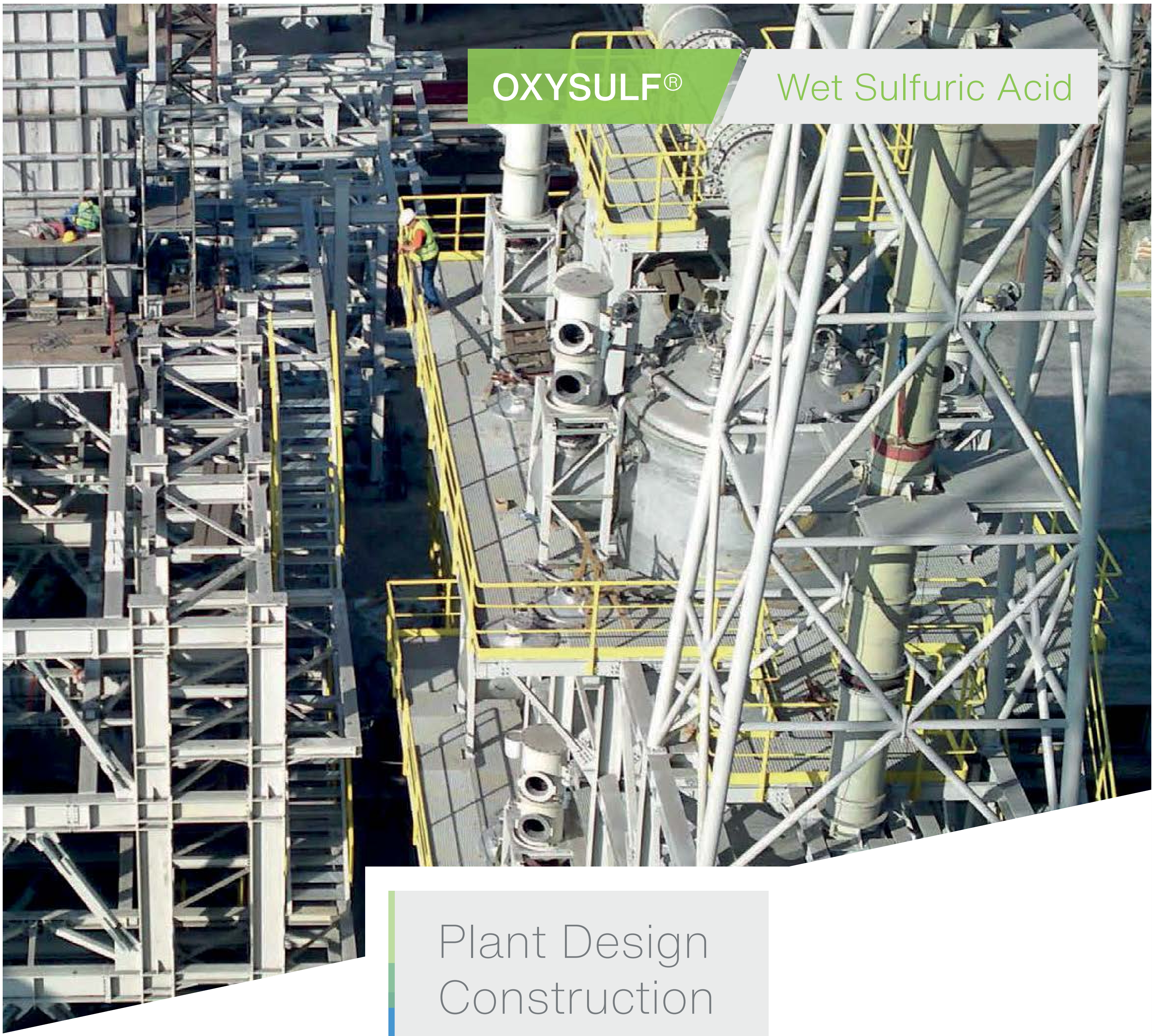
Using hydrogen as fuel is another option, although it may necessitate redesigns for furnaces to deal with hydrogen's corrosive nature. Biofuels are yet another option. While used cooking oil and algae tend to be what people think of when discussing biofuels, synthetic natural gas or biogas can also be an option for decarbonising refineries, replacing natural gas without requiring major modification to existing equipment.

As far as output goes, refineries in Europe and the US are likely to see an increasing share of biofuels blended into diesel. Use of used cooking oil (UCO) in biodiesel production has been increasing rapidly in the EU – its status as a waste feedstock has made it eligible for double counting in many member states. Also increasing in popularity as a feed is hydrotreated vegetable oil (HVO), the market for which is projected to double by 2025. Annex IX-A of the EU's Renewable Energy Directive (RED II), provides an overview of feedstocks that are eligible for double counting. Among these are palm oil mill effluent (POME), esterified into FAME biodiesel. Sludge palm oil (SPO) also seems to be a suitable feedstock for HVO production, where high free fatty acid content does not inhibit the production process, leading to less need for pre-treatment.

Is this a turning point?

The shake-out caused by the last year of low refining margins is accelerating changes already occurring in the industry. As refineries close in North America, Oceania and Europe and open in Asia, the Asia Pacific region is rapidly becoming the epicentre of world refining industry. The Middle East is also growing rapidly. Refining capacity in the Middle East is set to rise to 12 million bbl/d by 2023, up from 9.9 million bbl/d in 2019. Figure 1 shows new crude distillation capacity installation worldwide over the next few years. It has even been suggested that this ongoing shift in regional focus may ultimately lead to new oil price benchmarks being established beyond WTI and Brent. For example, Abu Dhabi has recently launched a new oil futures contract for its Murban crude rather than tying prices to Dubai levels.

Meanwhile, Atlantic basin refineries are looking increasingly to plastics and petrochemicals, biofuels and other lower carbon options to diversify away from a reliance on low margin crude conversion. Rationalisation of capacity in North America and Europe and new capacity in the Middle East and Asia will also change the dynamics of sulphur production from refineries. Almost all new sulphur recovery capacity in refineries over the medium term future will be in China, India and the Middle East. ■



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SulGas 2021

This year's SulGas conference, covering sulphur recovery and gas treating with a special focus on India and southeast Asia, was run as a virtual event from February 1st to 3rd 2021.

PHOTO: LINDE ENGINEERING INDIA

The Sadara Chemical Company complex, Al Jubail, Saudi Arabia.

SulGas, organised by Three Ten Initiative Technologies LLP, is India's only conference on sulphur recovery and gas treatment. This year, the conference's third, was forced to be run as a virtual event due to the continuing covid pandemic. However, that format saw attendance rise from last year's 170 to 266 delegates from 84 participating companies, 55 of those based outside India, including a number of major operators such as ADNOC, Pertamina, Qatar Petroleum, KNPC etc.

SRU operation

Dharmeshkumar Patel and Ahmad Nyeazi of Sulphur Recovery Engineering looked at the challenges inherent in operating SRUs at turndown, especially continuing to meet stringent environmental regulations, including design challenges; the effects of turndown on main/ancillary equipment and overall recovery efficiency; options and solutions for overcoming operational issues; and three case studies that looked at reaction furnace performance, converter issues, and the effects on the final condenser.

Uday Parekh of Blasch Precision Ceramics described how the performance of the SRU reaction furnace and thermal oxidiser can be enhanced by the careful deployment of internals to improve the overall combustion and deliver the desired conversion or destruction of the principal feed components and contaminants, respectively. This requires the customised calibration of the time, temperature and turbulence (the "Three T's" of combustion) in each of these combustion chambers.

Lastly, Ershad Hussain from Numaligarh Refinery Ltd gave an operator's-eye view of SRU operational issues. The refinery runs a small single-train sulphur recovery unit (<20 t/d) alongside the associated amine and sour water stripper (SWS) sections. Issues have included foaming in the amine system and hydrocarbon carryover in the SWS, and high pressure drop across the SRU. The presentation also looked at the bottlenecks of operating a single SRU train and innovations that have been adopted to reduce unit downtime.

Oxygen enrichment

Oxygen enrichment can enhance capacity enhancement of an existing SRU or reduce capex requirements for a greenfield SRU by reducing equipment and piping sizes and, as well as reducing opex by lowering TGTU solvent circulation and incinerator fuel consumption. Rahul Dole and Sohan Bhatt of Linde Engineering India described work that their company had conducted on the development and delivery of oxygen enrichment technologies over the past 25 years. Linde's SURE™ technology can be used from low to high level enrichment, up to 100% oxygen. The company can also supply associated technologies for oxygen generation such as a liquid-gas trailer plus vaporiser, VPSA or air separation unit.

Attila Racz and Mike Smeltink of Comprimo presented a case study of an oxygen enrichment project at the Hyundai Oilbank refinery in South Korea. The refinery had increased its crude oil and heavier distillate processing capacities, which resulted in a significantly higher sulphur processing requirement.

Comprimo worked with Hyundai Oilbank to determine the bottlenecks of their two ARUs, SWSs and SRUs, which required a capacity increase from about 380 t/d to 660 t/d. The most effective way to increase the capacity of the SRU was by using oxygen enrichment. By combining mid and high-level oxygen enrichment with upgrades in the ARUs and SWSs to

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limit hydrocarbons, the project was able to achieve an increase in processing capacity to 740 t/d.

Amine systems

Trending Tragedy – Why Amine Systems Fail was the topic for Philip le Grange of Sulphur Experts. Equipment malfunction or unplanned shutdown of an amine system can have a devastating effect on profitability. Philip aimed to identify the highest probability threats to a facility's operability and reliability, via an anaylsis of 400 cases of major amine system failure investigated by Amine Experts and other industry leaders over the last 20 years. These include detailed root-cause analysis related to corrosion, foaming, hydraulic restrictions and incorrect specification that have limited or crippled facilities the world over.

> **Biogas is often contaminated with toxic levels of hydrogen sulphide.**

Jenny Shao of Koch subsidiary Eco-Tec Inc looked at how normal operation of an amine circuit generates heat stable salts (HSS) as a result of feed gas and amine degradation. HSS cause corrosion of equipment, foaming, and reduced amine capacity. Eco-Tec provide an ion exchange-based HSS removal technology call *AmiPur®* which selectively removes HSS and recycles purified amine back into the circuit, allowing for consistent performance and reduced amine purchasing costs.

Tail gas treatment

Jochen Geiger and Anantha Kukkuvada of Ametek Process Instruments tackled how to ensure TGTU absorber performance to meet statutory requirements of 99.99% sulphur recovery, including how to monitor amine absorber efficiency and how to protect the amine from chemical reactions caused by process upset, by measuring the hydrogen and hydrogen sulphide concentrations downstream and upstream of the absorber. Downstream measurement includes the measurement of carbonyl sulphide and carbon disulphide, and what can be learned from this.

Sulphur removal

Biogas is often contaminated with toxic levels of hydrogen sulphide which can cause health and safety problems as well as corrode boilers, piping, and other equipment when mixed with carbon dioxide. Jenny Shao of Eco-Tec showcased the BgPur™ gas contactor, which removes up to 99% H_2S during biogas production, leading to significant cost savings through lower maintenance requirements and increased energy recovery and generating elemental sulphur as a by-product.

M. Shanmuga Sundaram and Sunder Parthasarathy of Süd-Chemie took delegates through the company's range of catalysts for sulphur removal, including SulfoMax® for wet-gas sulphur removal and the W-Series to treat acid gas from sour water strippers, Claus tail gas and SO_2 gas/spent acid.

ActiSorb GP® series adsorbents can remove sulphur from natural gas to meet pipeline specifications. Finally, HDMax® 230 is a low temperature multifunctional hydrogenation catalyst with high activity for tail gas treating units in SRUs (see page 41). It operates at an inlet temperature range of 210-220°C and can raise overall sulphur recovery levels to almost 100% depending on the type of sulphur species.

Process analysis

Anantha Kukkuvada of Ametek Process Instruments discussed installation guidelines and sample handling systems for the analysers used on modern sulphur recovery and tail gas treating units, along with industry best practice recommendations based on maintenance history records and direct experience. Analyser function and SRU/TGTU process control are well-understood at the design and process engineering levels, but sometimes less understood at the operational level. Good design, adherence to best practices, a comprehensive preventative maintenance program and process training are the keys to complete utilisation of analyser assets and improved operation.

Matt Coady of Delta Controls considered monitoring temperature in Claus thermal reactors. Reliable temperature instrumentation can be successfully retrofitted on existing reactors if proper consideration is taken in regards to details such as design, placement, and product selection.

Nathan Hatcher and Simon Weiland of Optimized Gas Treating, Inc. presented a tool for monitoring, optimising and troubleshooting gas treating and sulphur processing units. Integration of SulphurPro® and ProTreat® rate-based simulators has been achieved in a Microsoft Excel spreadsheet framework that permits ready and flexible integration with other OpCo and EPC software.

Sulphur handling

Sean Mathew of Controls Southeast considered strategies to minimise the cost of a piping thermal maintenance system. Thermal maintenance systems can utilise a broad range of technologies from bare tube tracing to fully jacketed pipe. However, regardless of the technology employed, engineering must be performed to design the system. The approach used can have a dramatic impact on the cost. Sean presented four strategies for minimising total cost: match the heating technology to the application; optimise the heating circuit lengths; optimise the utility infrastructure; and structure the bid process to reward optimisation.

Yves Herssens of DuPont Clean Technologies looked at tackling emissions from sulphur pits. The vapour space in sulphur pits contains H_2S, SO_2, and sulphur vapours that need to be vented. Depending on the concentrations, H_2S scavengers or packed tower caustic scrubbers have been typically applied to remove these sulphur contaminants from the vent streams. However, sulphur vapour quickly condenses to a fine elemental sulphur particulate, causing these systems to suffer from plugging. Yves described the use of DuPont's DynaWave® scrubber that removes sulphur contaminants without plugging.

Finally, Martin Joksch of P&P Industries AG described how recovery of sulphuric acid in a refinery spent acid recovery system can be achieved as a clear condensate at a conversion rate beyond 99.999 %. A catalytic triple stage process recovers the heat of oxidation, hydration and condensation.

Poster sessions

Poster sessions also included:

- Case studies of SRU troubleshooting;
- Flooding in a TGTU reactor;
- Ammonia vs caustic dosing in a TGTU quench tower;
- Automation of combustion control by an air demand analyser to maximise profitability and reliability from a sulphur recovery unit;
- Lean amine pre-filter system modification; and
- A case study of condenser plugging. ■

Treating highly sour gas

Sour gas processing presents a number of unique challenges, especially at high levels of hydrogen sulphide and carbon dioxide content.

Right: The Advantage Glacier sour gas plant, Canada.

PHOTO: EQUINOX ENGINEERING

Approximately 40% of the global natural gas reserves, either standalone or associated with oil production, are classified as 'sour', due to their content of either hydrogen sulphide (H_2S) or carbon dioxide (CO_2). Among these, about 30% (12% of total gas reserves) contain more than 10% CO_2, and 15% (6% of total gas reserves) contain more than 10% H_2S. Major H_2S-containing fields are present in Abu Dhabi (10-35% H_2S), Kazakhstan (18% H_2S), southern Russia (30% H_2S) and Alberta (ca 35% H_2S). Some sour gas also remains in France (15% H_2S). High CO_2-content gas can be found in Indonesia, Thailand and Malaysia (25-70% CO_2 content), and North America (20-65% CO_2). In all of these cases, the sour component must be removed before usable sales gas can be produced.

Treating sour gas

The main way of removing H_2S and CO_2 from gas streams is via solvent systems, either chemical solvents, physical solvents, or hybrids. Chemical solvents tend to be based on an amine blend, typically a variant on monoethanol amine (MEA), diethanolamine (DEA) or methyl diethanol amine (MDEA). Tertiary amines like MDEA require less reboiler duty, although it is less good at removing CO_2. Amines are typically are the lowest cost option for low to moderate levels of H_2S/CO_2 removal, and can also remove carbonyl sulphide (COS) from gas streams. They also typically have low solubility for hydrocarbons, which otherwise can affect downstream Claus processing. However, they have low selectivity for mercaptans, which may need further processing to remove, and require low pressure steam to regenerate the solvent, which can become expensive for high concentrations of acid gases. Using natural gas to generate the steam means that as acid gas concentrations increase, the proportion of sales gas generated can fall rapidly, with costs doubling every 20-25% increase in H_2S/CO_2 concentration. However, it is possible that these requirements could be met in other ways; at the 2019 SOGAT conference, ADNOC reported on feasibility studies of using solar power to regenerate solvent to free up more fuel gas for sale.

Physical solvents such as *Selexol* work purely by solubility in proportion of the partial pressure of the acid gas, with no chemical reaction. As a result the solvent circulation rate is not highly proportional to the quantity of acid gas removed. This means physical solvents can achieve higher acid gas loading at high acid gas concentrations than amine systems, and can be regenerated at lower cost. However, co-absorption of hydrocarbons is significant and can result in product losses, especially for higher chain components, which can restricts the use of physical solvents for many gas treating applications. As a result, there are hybrid systems like Shell's *Sulfinol* which blend the two types of solvents for specific applications, in such a way as to maximise the benefits and minimise the limitations of each type of solvent. Prosernat's *Sprex* process combined cryogenic separation with an 'energised' MDEA amine recovery section.

Other separation methods can be used, for example using fractional distillation via refrigeration. Fractionation can be smaller and simpler compared to a large gas treating system, but does require an energy intensive refrigeration system to condense the acid gas. ExxonMobil has developed a variant – the controlled freeze zone (CFZ) process, which can recover almost pure methane from a sour gas stream, suited for high acid gas concentrations (>30%).

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Different methods can be used in series to achieve the specific mix of gas component removal and/or recovery, for example to recover H_2S for Claus processing but reinject carbon dioxide for enhanced oil recovery.

Semi-permeable membranes can also be used for physical separation of gas components. Membrane systems are best suited for feed gas at high pressure and are most attractive when the CO_2 content of the gas is greater than 25%. At high H_2S concentrations the hydrogen sulphide can soften some membrane materials, but UOP have developed cellulose acetate membranes for high H_2S service.

Process or reinject?

Once the acid gas has been separated from the sales gas, the question then arises as to what to do with it. Claus recovery of sulphur from H_2S used to be the standard treatment method. However, prices for sulphur can be volatile and are often quite low. Storage of large volumes of sulphur is not always a practical option, especially in a region like Central Asia where producing sites may be distant from markets. Emission of carbon dioxide from the acid gas is also becoming increasingly problematic as carbon pricing and emission limits continue to spread. Consequently there is an increasing trend towards reinjecting the acid gas back into the well or other suitable site, potentially generating additional reservoir pressure and possibly assisting with associated oil recovery, where present. Of course, acid gas injection (AGI) requires a geologically suitable site nearby for the gas to be injected into. It also may offer potential problems when the AGI system is down for whatever reason – flaring of the acid gas may not be possible due to local regulations, and the entire gas plant would therefore need to be shut down.

It is theoretically possible to recover some of the energy of the H_2S by burning it and converting it to SO_2 before reinjection, but the feasibility of such a process remains to be demonstrated at large scale.

Health and safety considerations

Handling large volumes of hydrogen sulphide offers the possibility of deadly leaks. A comprehensive risk management strategy must therefore form part of any large scale sour gas operation. This can put a particular focus on layout choices for the sour gas facility. At ADNOC's Shah sour gas plant in Abu Dhabi, for example, this meant separating the well heads from the main working areas by a couple of kilometres. However, this of course means longer pipe racks and therefore more expense.

Reliability of equipment, especially seals, also becomes paramount, as well as monitoring and maintenance of the plant.

Corrosion

H_2S and CO_2 are acidic when mixed with water, as the name acid gases suggests. High partial pressures and higher water content can lead to severe corrosion of steel pipework, and can lead to catastrophic leaks. The experience of the Kashagan project in Kazakhstan shows the time and expense that such corrosion can cause. The highest grade steels must be used for acid gas pipework, which can increase the expense of an operation, and there must also be constant corrosion monitoring. ■

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Sulphur recovery using WSA technologies

Wet sulphuric acid technology when used as tail gas treatment for a Claus unit, or in place of a Claus unit with amine-based tail gas unit, brings many benefits if there is a market for the sulphuric acid and steam. A new alternative is to have a WSA unit in combination with a Claus unit and to recycle the acid to the Claus unit for higher sulphur recovery efficiency.

Most new oil and gas field discoveries today are sour and require a certain amount of cleaning up as part of the process to bring products to market. Sulphur continues to be a significant byproduct from oil and gas upgrading in both refining, gas plants and various other processes. Although sulphur is a product in its own right, for many in the oil and gas industry, it is regarded as a waste product.

Sulphur emissions standards for hydrocarbon products and in general continue to tighten, covering emissions from refineries, gas plants and production facilities. For example, current World Bank standards prescribe that Claus installations in refineries should emit no more than 150 mg/Nm³ sulphur dioxide, corresponding to a sulphur removal efficiency of 99.9+%.

Operators are striving to meet emission standards at minimum capital and operating costs by using optimised technology, but are also looking to reduce their carbon footprint. Unfortunately, a reduction in carbon footprint doesn't go hand in hand with a reduction in sulphur emissions and it's the last few points of a percentage for higher SRE where the costs escalate.

In order to reach sulphur recovery efficiencies of 99.9+%, a very effective tail gas treatment unit (TGTU) is required downstream of the Claus unit. The Claus process with conventional tail gas treatment typically comprises a two-stage catalytic Claus plant combined with a hydrogenation and amine-based tail gas treating unit, sometimes referred to as a BSR/amine plant, or SCOT plant. The TGTU hydrogenates all remaining sulphur compounds to H_2S and then uses an amine system to capture and recycle the H_2S.

Amine-based TGTUs are relatively expensive to install as well as to operate, and involve high energy consumption for amine regeneration and final H_2S incineration. This leads to high compliance costs and unfavourable CO_2 footprints.

There have been incremental improvements to the amine-based tail gas treating processes over the years, as well as improvements to the amines used, e.g. increased H_2S selectivity often using proprietary amines, but the process remains largely the same.

Alternative technologies are available that offer lower capex and opex. One example is SuperClaus technology from Comprimo, part of the Worley Group, which can be coupled with caustic scrubbing to achieve very low sulphur emissions. Capex is lower, but there is an additional waste stream, mainly sodium sulphate in water, which has associated disposal costs. This option may be suitable for smaller plants (up to 300 t/d), but is not ideal for larger plants.

Haldor Topsoe's Wet gas Sulphuric Acid (WSA) process also offers an alternative tail gas treating technology. The WSA process dates back to 1980 and around 165 units have been licensed.

TopClaus® combining Claus and WSA technologies

The new TopClaus® technology developed by Haldor Topsoe and Comprimo combines Claus and WSA technologies (Fig. 1). Using a WSA tail gas unit with acid recycling reduces energy intensity but still maintains the sulphur recovery in excess of 99.9%. Equipment count, capex and carbon footprint are all reduced. The product is still sulphur, but with enhanced operational benefits.


PHOTO: COMPRIMO/TOPSOE

Topsoe WSA unit.

In the TopClaus® process, sour gas is treated in a simple conventional two-stage Claus unit with 95-97% SRE to produce elemental sulphur. The tail gas from the Claus unit proceeds to the WSA unit, where the remaining sulphur compounds are removed to yield a total SRE of more than 99.9%.

The small amount of sulphuric acid produced is then returned directly to the thermal reactor of the Claus unit to be reprocessed to elemental sulphur. This makes it possible to have all sulphur product in the form of elemental sulphur. It is

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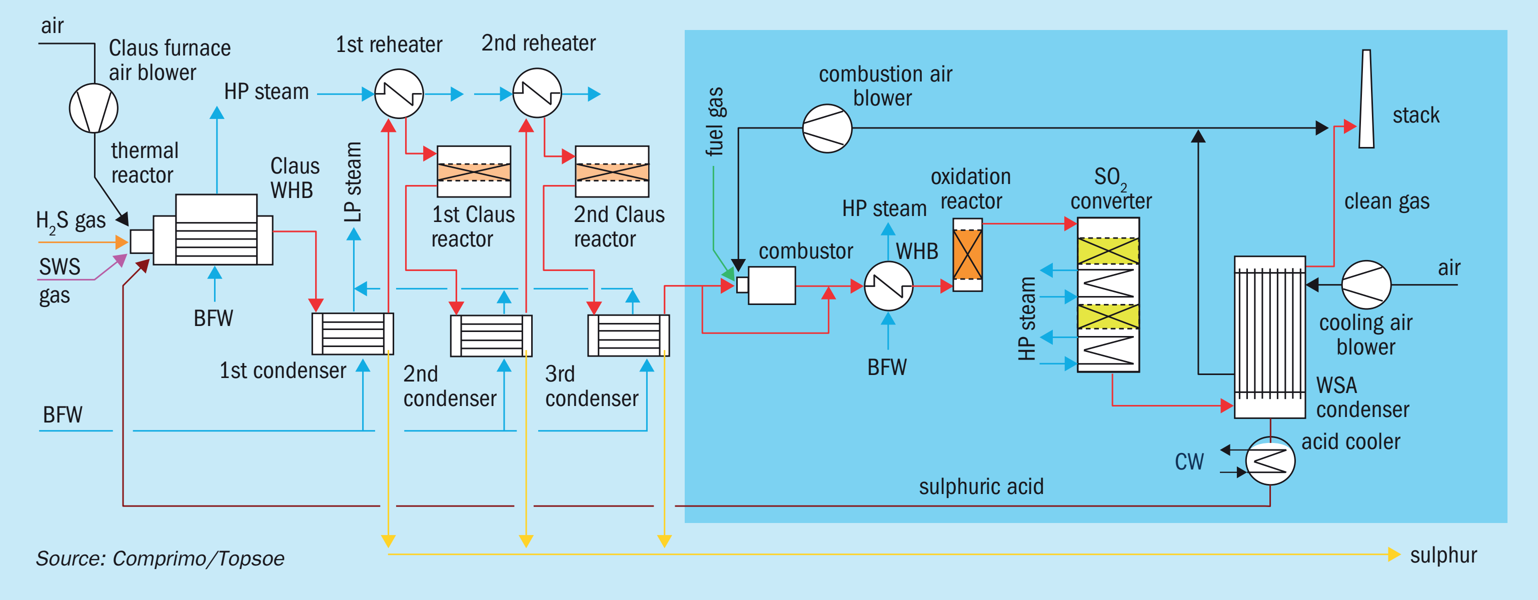
Fig. 1: **TopClaus® simplified process flow diagram**



Source: Comprimo/Topsoe

also possible to extract more or less of the sulphuric acid as product if there is a need for it locally.

Sulphuric acid is a very effective oxygen carrier, which means the size of the Claus unit can be reduced compared to a Claus unit with a traditional amine-based TGTU, thus significantly reducing TopClaus® capex.

The following reactions compare the amount of dilution gas in the overall Claus reaction with air, pure oxygen and sulphuric acid:

Overall Claus reaction with air

$$4H_2S + [2O_2 + 8N_2] \rightarrow 4S + 4H_2O + 8N_2$$

12 moles dilution gas

Overall Claus reaction with pure oxygen

$$4H_2S + 2O_2 \rightarrow 4S + 4H_2O$$

4 moles dilution gas

Overall Claus reaction with sulphuric acid

$$3H_2S + [H_2SO_4 + 0.4H_2O] \rightarrow 4S + 4.4H_2O$$

4.4 moles dilution gas

As shown in the reactions above, in the overall Claus reaction with air there are 12 moles of dilution gas. Using pure oxygen instead of air removes the nitrogen leaving just four moles of dilution gas. Finally, recycle acid is an excellent oxygen carrier and leads to only 4.4 moles of dilution gas and so also facilitates reduction of air demand by about 20%. In addition, compared to a conventional TGTU there is no CO_2 in the recycle stream to the Claus thermal reactor.

The Claus thermal reactor chemistry with sulphuric acid (H_2SO_4) has been demonstrated by Alberta Sulphur Research Ltd (ASRL) and verified by dynamic CFD modelling by HEC Technologies:

H_2SO_4 (droplet)
↓ fast
H_2SO_4 (gas)
↓ instantaneous
$H_2O + SO_3$
↓ fast
$H_2O + SO_2 + ½O_2$
standard Claus components

Table 1 compares the equipment count for an amine-based TGTU with a WSA-based TGTU (TopClaus®).

As shown in Table 1, the WSA-based TGTU has nine fewer items of equipment, but perhaps the most pertinent is the removal of the three columns, a good indication of lower capex for the WSA-based TGTU.

TopClaus® case study

To quantify the capex and opex savings of TopClaus®, consider the following case study for a typical greenfield oil refinery SRU with a capacity of 270 t/d treating typical refinery feed streams of amine acid gas and sour water stripper off gas. A sulphur recovery of 99.9% was required and TopClaus® was compared with conventional design. An acidified MDEA was used for the conventional design so the benefits of that over generic MDEA were already accounted for. A Class 4 Factored Estimate was prepared on a US Gulf Coast basis (see Table 2).

The cost estimate considered facilities only within the SRU (ISBL), based on 8,500 operation hours per year. Common factors were excluded, for example, site preparation. Sulphur product value was also excluded as it was common for both configurations.

Table 1: **Tail gas treatment equipment count comparison**

Equipment type	Amine-based TGTU	WSA-based TGTU
Furnaces/burners	1	1
Columns	3	0
Reactors/converter	1	2
Vessels	4	2
Tanks/sumps	2	1
Heat exchangers	7	8
Blowers*	1	2
Pumps*	6	2
Filters	3	0
Package units*	0	1
Total equipment	**28**	**19**

**excluding installed spares*

Source: Comprimo/Topsoe

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Table 2: **Case study cost basis**

Item	**Cost/value, $/unit**
HP/MP steam, t	12.00
LP steam, t	9.00
Boiler feed water, t	2.00
Boiler blow down, t	1.00
Cooling water, m^3	0.04
Natural gas, Nm^3	0.25
Electric power, kWh	0.10

Source: Comprimo/Topsoe

Table 3: **Case study: cost and value of utilities**

	Claus + amine	**TopClaus®**	**Claus + amine**	**TopClaus®**
Consumption	**consumption/hour**		**USD/year**	
BFW, t	33	50	561,000	850,000
Electric power, KWh	1,900	1,500	1,615,111	1,275,000
Natural gas, Nm^3	200	440	425,000	935,000
Cooling water, m^3	0	15	0	5,000
Total consumption			**2,601,000**	**3,065,000**
Production	**production/hour**		**USD/year**	
HP/MP steam, t	32	41	3,264,000	4,182,000
LP steam, t	0.3	8.7	33,000	666,000
Boiler blow down, t	0.8	1.1	7,000	9,000
Total production			**3,304,000**	**4,857,000**
Net operating income			703,000	1,792,000

Source: Comprimo/Topsoe

Table 4: **Case study: capex/opex comparison**

	Claus + amine	**TopClaus®**
Capex estimate, USD	69,400,000	61,800,000
Operating income, USD/year	703,000	1,792,000
Maintenance, USD/year	1,388,000	1,236,000
Catalysts and chemicals, USD/year	200,000	200,000
Costs over 10 years, USD	78,250,000	58,240,000

Source: Comprimo/Topsoe

The results were striking and are detailed in Table 3, covering utility production/consumption, and then monetised in Table 4 to give capex and opex over ten years.

TopClaus® consumes more natural gas, BFW and cooling water, but this is almost offset by the reduction in use of electric power, i.e. $3.0 million per annum vs $2.6 million pa for all consumptions. On the production side, TopClaus® produces much more steam giving an overall production figure of $4.9 million pa vs $3.3 million pa. Overall, operating income for TopClaus® is just over $1 million pa greater than that for the conventional design.

The capex for the TopClaus® configuration is $61.8 million vs $69.4 million, giving a saving of 11%, (and up to 30% if generic MDEA were used in the conventional technology), add to this opex savings over ten years, this gives a saving of $20 million.

TopClaus® achieves a sulphur recovery equivalent to the conventional design, it's a simpler process with fewer parameters to control, and the economic benefits are substantial. The Claus unit itself will be smaller due to the recycle and oxygen carrying capacity of the acid.

Additional flexibility can be provided by producing a small sulphuric acid stream, which could be useful in a refinery application, although there would need to be special circumstances to progress this route. Maintaining a single product stream is preferable in virtually all applications.

Brownfield application, i.e. revamps, provide unique opportunities, with perhaps limited space, a need for marginal increased capacity, and a requirement to meet increasingly tight emissions specifications. The WSA section usually does not require additional manpower for operation; it is compact, and it requires minimum shutdown time to incorporate.

Application to natural gas plant SRUs also affords some enhanced benefits for TopClaus®:

With a conventional TGTU:

- significant CO_2 in feed reduces furnace temperature and increases size of conventional TGTU;
- CO_2 co-absorbed in TGTU absorber is recycled to inlet of Claus unit;
- TGTU amine requires leaner loading to allow CO_2 "slip".

With TopClaus:

- all CO_2 passes through to stack;
- smaller recycle → smaller SRU.

In summary, the key benfits of TopClaus® are:

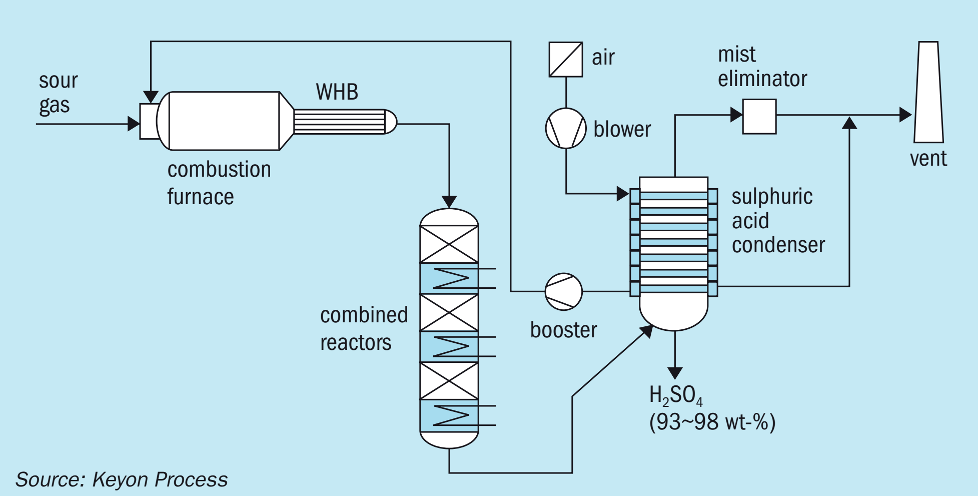
- simple, robust and efficient combination of proven technologies with integration demonstrated by ASRL;
- >99.9% sulphur recovery;
- all sulphur product in the form of elemental sulphur, with the option of sulphuric acid for use or sale if desired;
- smaller Claus unit due to sulphuric acid recycle;
- no waste streams;
- suitable for greenfield projects as well as revamps;
- high thermal efficiency with HP steam export;
- 10-30% lower capex compared to Claus unit with amine-based TGTU;
- no energy required for regeneration of amines and tail gas incineration, resulting in significantly lower opex, leading to a more attractive NPV and also reduces CO_2 footprint.

ECOSA® as an alternative solution to Claus

ECOSA® is a wet sulphuric acid technology widely applied in different industries, including but not limited to refineries, coal gasification, metallurgical, coal coking, and viscose fibre production. A typical flow diagram for the ECOSA® process is shown in Fig. 2.

To date, more than 40 ECOSA® reference projects have been awarded to Keyon Process, most of them are functioning as an alternative solution to the Claus process, providing a number of benefits due to the following:

Fig: 2: **Typical ECOSA® process**



Source: Keyon Process


PHOTO: KEYON PROCESS

Yatong 450 t/d ECOSA® plant.

Table 5: **Recent ECOSA® projects**

Project	Production (t/d)	Industry
1	450	refinery
2	550	viscose fibre
3	60	coal chemical
4	35	coal chemical
5	350	fine chemical
6	80	coal chemical
7	39	coal chemical
8	34	coal chemical
9	50	coal chemical
10	60	coal chemical
11	90	coal chemical

Source: Keyon Process

- Since the Emission Standard of Pollutants for Petroleum Chemistry Industry (GB 31571) and the Emission Standard of Pollutants for Petroleum Refining Industry (GB 31570) were promulgated in the year 2015, the tail gas emission requirements in China have become much more stringent. $SO_2 \leq 100$ mg/Nm³, NOx ≤ 50 mg/Nm³ and acid mist ≤ 5 mg/Nm³ are commonly required.
- Claus technology is listed as a key regulated hazardous chemical process by the Chinese government. A standby spare plant is normally required which increases the capex to more than a single ECOSA® plant.
- The Chinese government restricts coal consumption in coastal areas and chemical plants, especially refinery plants, have a strong demand for high quality superheated steam. ECOSA can therefore bring the client practical benefits.

Table 5 shows a selection of projects from the past three years where ECOSA® was chosen over Claus.

The main reasons ECOSA® is preferred are:

- greater steam production;
- sulphuric acid in local demand;
- cleaner emissions;
- capex and opex.

The first two projects listed are described below.

Yatong ECOSA® project

In 2017, Yatong refinery, located in the Shandong province of China, chose ECOSA® instead of Claus to be used in the sulphur recovery unit for its brand new 3 million t/a heavy oil hydrocracking plant.

The 150,000 t/a (450t/d) new wet sulphuric acid ECOSA® unit, capable of treating both the sour gas from the hydrocracking process as well as regenerating 2.5 t/h spent acid discharged from the alkylation plant in the same refinery, has been running smoothly since early 2018 with an annual on-stream rate of more than 95%.

The reason Yatong refinery chose the wet sulphuric acid process instead of Claus for this project was mainly because the refinery had an existing 15,000 t/a Claus + SCOT plant running since 2010, with the following problems and demands:

After a few years of operation, the hydrogenation unit of the SCOT unit was running at low efficiency and channelling in the catalyst bed had formed shortcuts for the SO_2 flow which led to sulphur forming in the downstream equipment, blocking the strainer on the inlet line of the pump, which is located by the quench tower of the SCOT unit.

The SO_2 content in the SCOT off-gas was around 700-800 mg/Nm³, far greater than China's latest emission requirement of 100 mg/m³ in China.

The refinery plant has increased demand for high quality superheated steam. In addition, the client has an alkylation plant which requires fresh H_2SO_4 as raw material.

Since 2018, the 450 t/d ECOSA® unit has brought the following benefits to the owner:

- The 924 t/d (400°C, 4.0 MPa) steam production capacity of the ECOSA® unit is nearly double that which could have been provided by a similar scale Claus unit.
- The ECOSA® unit achieves clean tail gas emissions: $SO_2 \leq 100$ mg/Nm³, NOx ≤ 50 mg/Nm³, acid mist ≤ 5 mg/Nm³.
- Spent acid discharged from the refinery alkylation plant is regenerated to fresh H_2SO_4 by the same ECOSA® unit.

Jinkun ECOSA® project

A more recent case is the Jinkun chemicals ECOSA® plant, a sulphur recovery unit to treat H_2S sour gas emitted from the main CS_2 plant located in Hebei province. The plant has a production capacity of 180,000 t/a (550t/d) sulphuric acid and the operation of the ECOSA® unit has been running smoothly since early 2020 without shutdown.

The owner chose wet sulphuric acid technology rather than Claus to build this fairly large-scale sulphur recovery plant, mainly because sulphuric acid product is an essential chemical raw material for the local industry, and nearby factories have a strong demand for steam.

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PHOTO: KEYON PROCESS

Jinkun 550 t/d ECOSA® plant.

Since the start-up of the unit in early 2020, there was a period when there was not enough H_2S produced from the upstream plant, so the owner used sulphur and low pressure steam as feed to produce fresh concentrated sulphuric acid and high quality superheated steam. The operation lasted for about half a year and brought continuous benefits. It also proves that ECOSA® is adaptable to different sulphurous raw materials. The Jinkun 550 t/d ECOSA® plant has a steam production capacity of 1,100 t/d (@400°C, 4.0 MPa), with SO_2 ≤ 100 mg/Nm³ and acid mist ≤ 5 mg/Nm³ in the final tail gas.

Table 6: **Key benefits of ECOSA® over Claus + SCOT**

	ECOSA®	**Claus + SCOT**
Sulphur recovery efficiency, %	~99.9	~99.9
SO_2 in tail gas, mg/Nm³	$\leq$100	~300
Product type	H_2SO_4, 98 wt-%	sulphur
Steam production, t/t H_2S	~6 @ 4.0 MPa, 420°C	~2 @ 4.0 MPa, saturated
Operation difficulty	easy	difficult
Waste liquid discharge	no	yes, weak acid from quench
Utility consumption	lower	higher
Fuel gas consumption	no	yes

Source: Keyon Process

Table 7: **Projects of ECOSA® as tail gas treatment for Claus**

Project	**Production (t/d)**	**Industry**	**Main reasons ECOSA® is preferred over Claus**
1	150	coal chemical	• sulphuric acid in demand • extra steam production • cleaner emission
2	82	coal coking	
3	90	viscose fibre	

Source: Keyon Process

Table 8: **Comparison between Claus + ECOSA® and Claus + SCOT**

KPI	**Claus + SCOT**	**Claus + ECOSA®**
SO_2 in final tail gas, mg/Nm³	~300	$\leq$100
Product type	sulphur	sulphur + H_2SO_4
Process complexity	complex	simple
Operation difficulty	difficult	easy
Maintenance	difficult	easy
Site cleanliness	prone to sulphur leakage	clean
Weak acid waste	yes, from quench	no
H_2 consumption	yes	no
Fuel gas consumption	yes	no, only for start-up
Steam consumption	yes	no

Source: Keyon Process

Comparison between ECOSA® and Claus + SCOT

Table 6 shows the key benefits of ECOSA® compared to Claus + SCOT.

As shown in the table, compared to the Claus + SCOT process, the wet sulphuric acid ECOSA® process is simpler in process flow, lower emissions, smaller footprint, better operability with no secondary pollution. Moreover, Claus + SCOT consumes fuel gas and hydrogen during normal operation, while ECOSA® does not.

The main economic advantages of ECOSA® over Claus + SCOT are twofold:

- Better for energy conservation – the ECOSA® process generates a larger amount of high-quality steam (generally, @4.0MPa, 400°C) compared to an equivalent Claus + SCOT unit.
- Lower operational cost, mainly because ECOSA® process does not consume utilities such as hydrogen, fuel gas and steam, which are always required in Claus + SCOT units.

ECOSA® as tail gas treatment unit for Claus process

Besides the projects in which the ECOSA® wet sulphuric acid process has fully replaced the Claus process, Keyon Process currently has three ECOSA® projects in the development stage, in which ECOSA® will be used as a tail gas treatment unit for the upstream Claus units (see Table 7).

Feihong ECOSA® project

Feihong is a coal coking company in the coal-rich Shanxi province. The Company has an existing 70,000 t/a Claus + SCOT plant that uses H_2S sour gas coming from the Rectisol unit to produce elemental sulphur.

Due to the problems of high SO_2 emissions and sulphur blocking in the existing Claus + SCOT plant, the company has decided to revamp the Claus tail gas treatment unit, and has made a decision to build a new 82 t/d ECOSA® unit downstream of the Claus unit, in order to fully replace the old SCOT unit, meanwhile utilising Claus tail gas to produce sulphuric acid.

The sulphuric acid product will mainly be used by the waste water treatment unit at the site, for adjusting pH values of waste water. And the estimated 160 t/d steam @400°C, 4.0MPa by-product of ECOSA® will converge with the site steam supply network, saving a considerable amount of energy consumption.

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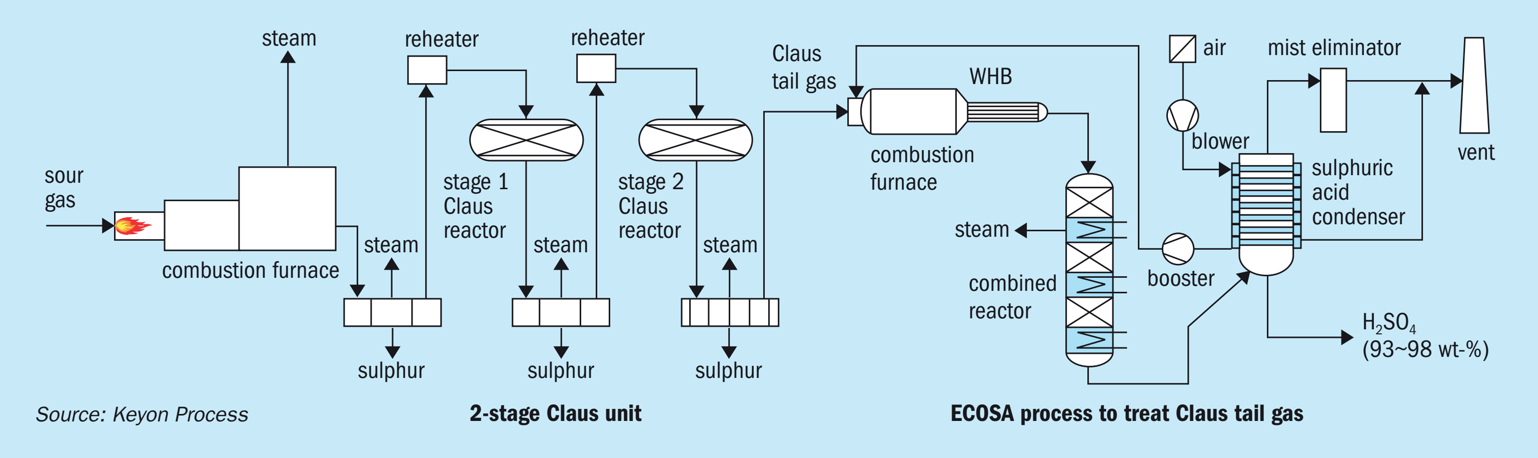
Fig. 3: **ECOSA® process treating Claus tail gas**



Source: Keyon Process

Claus + ECOSA® advantages

When applied as a supplementary tail gas treatment unit for Claus (Fig. 3), ECOSA® unit has a number of advantages over SCOT unit in terms of SO_2 emission, nil consumption of hydrogen and steam, plus producing sulphuric acid and steam (see Table 8).

Oxysulf technology with and without Claus

Oxysulf technology is an innovative wet process from Kanzler Verfahrenstechnik GmbH (KVT Technology) to produce sulphuric acid from off-gases containing sulphur-bearing compounds.

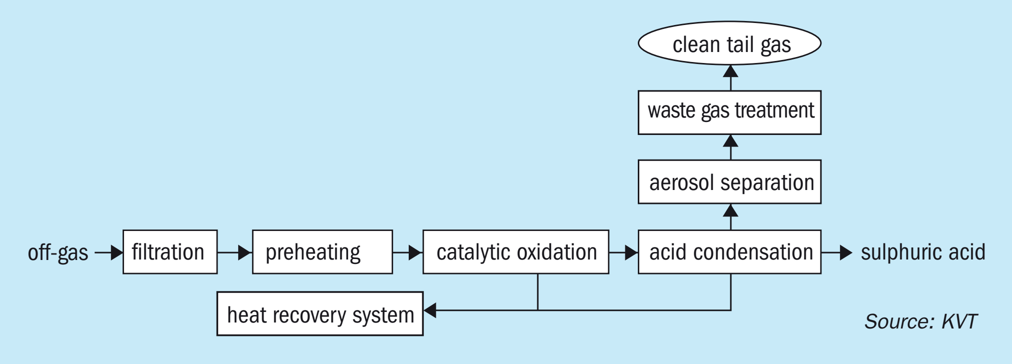
Oxysulf has the same chemistry as other wet sulphuric acid processes. Sulphur trioxide gas in the presence of steam forms gaseous sulphuric acid that is condensed by cooling to form liquid sulphuric acid. Depending on the concentration the acid dewpoint is typically 180-250°C.

$$SO_3(g) + H_2O(g) \rightarrow H_2SO_4(g) \ @ > \sim 250°C$$

$$H_2SO_4\ (g) \xrightarrow[\text{cooling}]{} H_2SO_4\ (liq) \ @ \ < 250°C$$

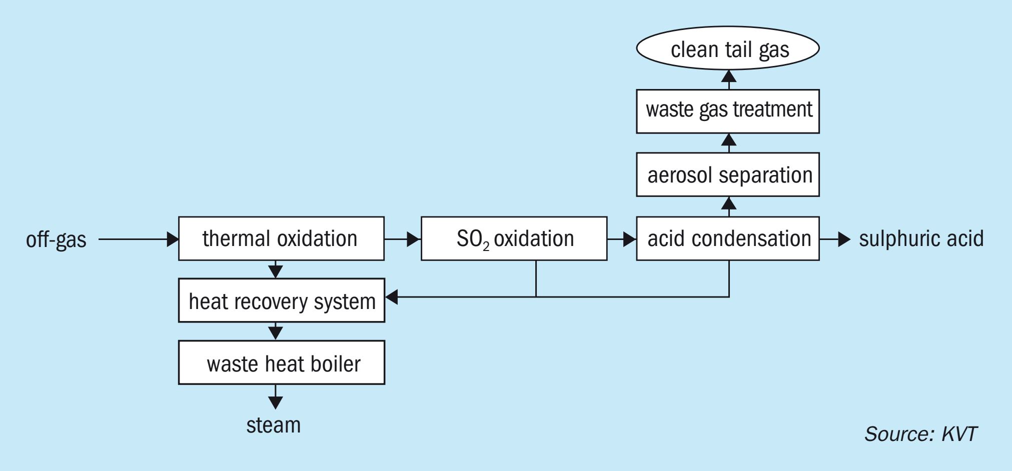
However, what differentiates Oxysulf from other processes is the mechanics it uses. In other technologies mist generation and separation take place inside glass tubes and the conversion rate is increased by double condensation, which requires cooling and reheating of the gas stream. By contrast, in KVT Oxysulf technology mist generation takes place in a concentration column with mist separation in a WESP. The conversion rate is increased by the tail gas reactor which operates at the same temperature as the WESP, which means no heating is required.

Fig. 4: **Oxysulf NK for lean gases**



Source: KVT

Fig. 5: **Oxysulf HK for strong gases**



Source: KVT

Key benefits of the Oxysulf technology include:

- Sulphuric acid produced as a second product ready for use in other processes;
- Oxysulf replaces further tail gas treatment;
- $SO_2 < 50$ mg/Nm³; H_2SO_4 aerosol < 3 mg/Nm³
- high overall conversion of sulphur components up to 99.9%;
- lean sulphur-containing streams can be fed to the process to increase the efficiency (e.g. from sour water stripping);
- no further consumables are required like NaOH, peroxide etc.;
- high energy efficiency.

Oxysulf has different plant configurations depending upon the feed type (clean or dirty, liquid or gaseous), feed composition (type and concentration of sulphur compounds e.g. H_2S, CS_2, COS, SO_2) and emission requirements.

In cases where concentrated sulphuric acid is requested Oxysulf may be used as tail gas treatment after the Claus process (see Fig. 4) or may even be used as a replacement to the Claus process (Fig. 5). Alternatively, if Oxysulf is used in combination with the Claus process and sulphuric acid is not required it can be retrofitted into the Claus process.

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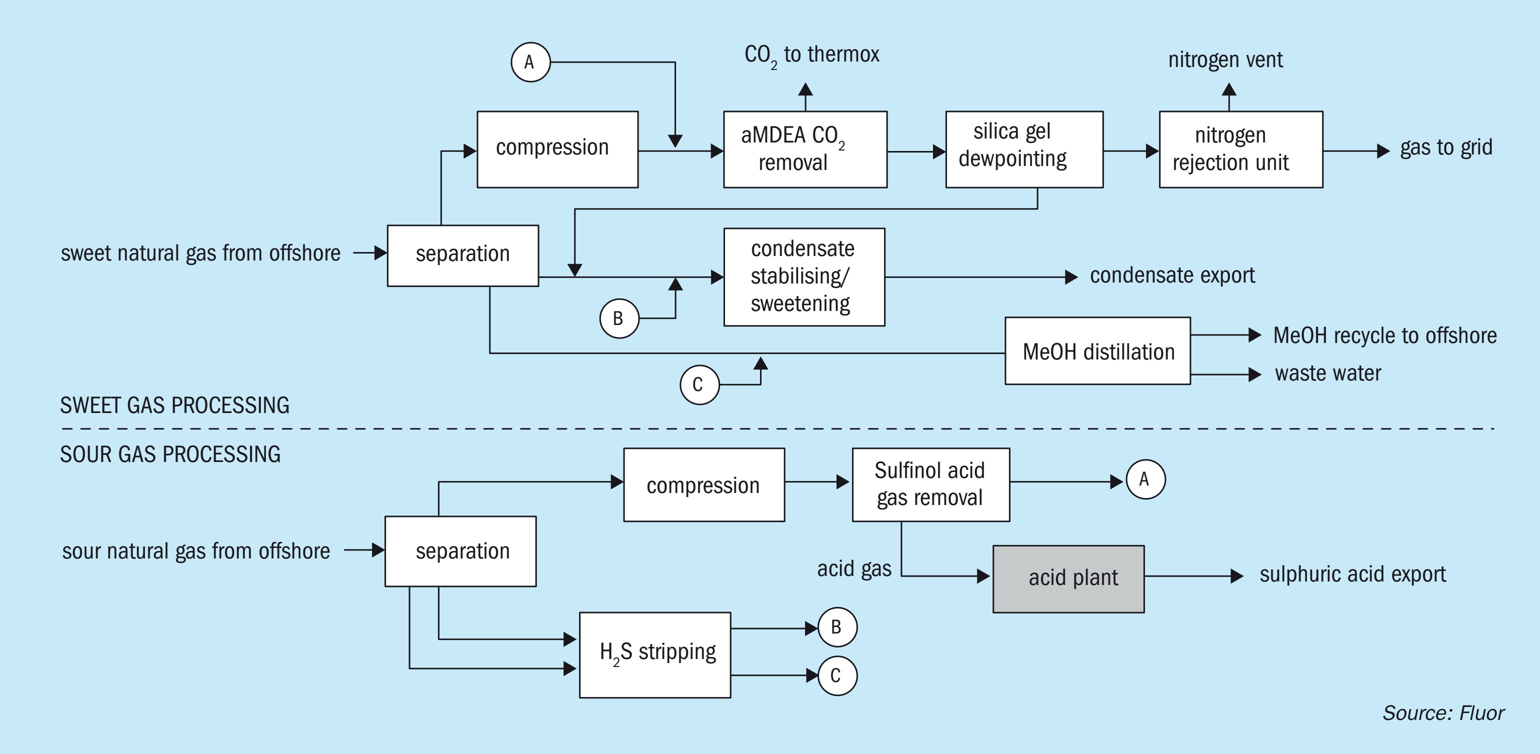
Successful start-up of a new WSA plant

M. Baerends of Fluor reports on the conception, engineering, construction, commissioning and start-up of a new sulphuric acid plant that replaced an existing acid plant at a European sour gas processing terminal. This highly complex gas processing facility handles sour gas from an off-shore field, containing H_2S that must be removed to meet transmission grid specifications. Various issues encountered, their resolution by the joint owner, Fluor and Topsoe team, and plant operating results are discussed.

Fig. 1: **Gas processing terminal scheme**



Source: Fluor

Overview of gas terminal

The gas terminal that is the subject of this article processes raw gas that is rich in both CO_2 and nitrogen. This terminal was among the most complex in Europe when built, and includes compression, an activated MDEA (aMDEA) system for CO_2 removal, a silica-gel type dewpoint bed system for water and heavier hydrocarbon removal, and finally a cryogenic nitrogen rejection unit (NRU) with associated deep dehydration and product gas recompression.

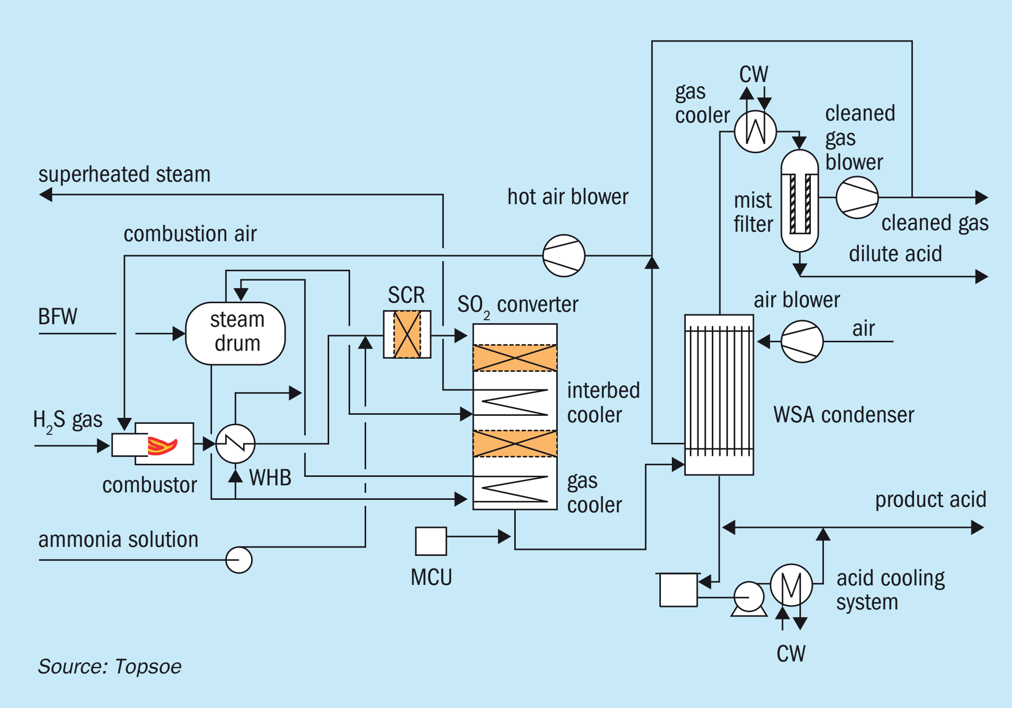
Further facilities include condensate stabilisation and caustic-based condensate sweetening, as well as a methanol distillation system for recycling methanol to the natural gas production fields (methanol being used as hydrate inhibitor).

The terminal includes two processing trains, one for sour gas processing and the other for sweet gas processing. Fig. 1 shows the two trains, and how they are linked.

Focusing on the sour gas processing train, feed gas enters a large separator-type slug catcher with overhead gas compressed by gas-turbine driven compressors from about 40 to about 80 bar. The compressed gas undergoes H_2S and mercaptan removal by means of Sulfinol treatment. As Sulfinol is a 'hybrid' chemical/physical solvent, it is well-suited to remove organic sulphur components such as mercaptans along with H_2S. Treated gas from the Sulfinol absorber is then routed to the sweet gas processing train where it enters upstream the aMDEA unit, subsequently passing through the silica-gel dewpoint beds and NRU systems.

Any liquids from the slug catcher are let down in pressure to a separator vessel

Fig. 2: **WSA overview scheme**



Source: Topsoe

in which hydrocarbon and water/methanol phases are separated, each liquid stream being directed to stripper columns in which H_2S is removed by fuel gas stripping. Overhead gas from these columns, together with flash gas from the Sulfinol system rich flash drum, is recompressed into the suction of the main gas compressors mentioned above.

Aqueous methanol liquor is routed to the water/methanol distillation in the 'sweet' gas processing train. Hydrocarbon condensate is also routed to sweet gas processing train for stabilisation and sweetening.

Acid gas from the Sulfinol regenerator is sent to the subject acid plant for conversion into sulphuric acid of 96 wt% ±2 wt-% concentration for export.

The utility system that supports the gas processing terminal is briefly described in so far as it is relevant to the project. The sour gas processing train has its own dedicated steam system, with steam generated primarily in the acid plant waste heat boilers, and consumed in the Sulfinol Unit for reboiling. Any excess steam is condensed in a trim condenser. Condensate from the trim condenser and Sulfinol reboiler is returned to a deaerator, augmented with demin water make-up, and pumped as BFW to the acid plant. An auxiliary boiler for start-up is also provided.

Acid replacement project

Engineering

Historically, the sour gas processing train operation, although generally successful, experienced issues related to the existing acid plant reliability. Eventually this led to a decision to replace the existing acid plant.

Fluor Ltd was contracted in to prepare a technology selection study for a replacement plant. The outcome of the study led to selection of the Wet gas Sulphuric Acid (WSA) process from Haldor Topsoe A/S. This process has been well proven in a range of applications and industries, from viscose production to metals smelting, coke oven gas, refineries and natural gas facilities.

Although a Claus-type sulphur recovery unit, followed by a tail gas treating unit (to achieve similarly low SO_2 emissions) would have been a possible alternative, this would have required new infrastructure for exporting liquid sulphur, while an existing product acid export storage tank with truck loading facilities was already available. Furthermore, the acid gas design composition was very rich in hydrocarbons (including aromatics), in line with the use of a 'hybrid' physical/chemical solvent designed to remove mercaptans as well as H_2S. Processing an acid gas so rich in hydrocarbons (and also containing large amounts of CO_2) in a Claus-type sulphur recovery unit might have been problematic. Finally, the capacity would have been on the lower end of capacity for a Claus unit.

Fig. 2 shows a flow diagram of the process scheme for the WSA process.

Acid gas from the Sulfinol regenerator is combusted with an excess of combustion air in the refractory-lined combustor.

$$H_2S + 1½O_2 \rightarrow SO_2 + H_2O \qquad (1)$$

$$\text{hydrocarbons} + O_2 \rightarrow CO_2 + H_2O \qquad (2)$$

After heat removal by 50 bar steam generation in the waste heat boiler (WHB), a small amount of ammonia solution is injected into the process gas prior to a selective catalytic reduction (SCR) DeNOx step in which NOx is catalytically reduced to N_2. Gas from the SCR reactor enters the 2-bed SO_2 converter in which SO_2 is converted to SO_3 by means of a proprietary vanadium pentoxide (V_2O_5) catalyst.

$$SO_2 + ½O_2 \rightarrow SO_3 \qquad (3)$$

The inlet temperature to the second bed is controlled using a coil that superheats saturated steam; the outlet temperature is lowered by means of another WHB at the converter outlet. In this WHB a large part of the SO_3 reacts with water vapour to produce sulphuric acid vapour.

$$SO_3 + H_2O \rightarrow H_2SO_4\ (g) \qquad (4)$$

Gas from this WHB enters the proprietary WSA condenser in which the gas is cooled in glass tubes with cooling air on the shell side. Sulphuric acid condenses in the tubes and drops out to the acid vessel.

$$H_2SO_4\ (g) \rightarrow H_2SO_4\ (liq) \qquad (5)$$

Condensation without excessive mist formation is promoted by the mist control unit (MCU) which adds a controlled amount of small particulates (condensation nuclei) to the gas. The concentration of particulates in the gas is important as both too high or too low concentrations of particulates in the gas can result in excessive mist formation.

The acid vessel, into which hot acid from the WSA condenser is drained, is cooled by means of acid circulation through a heat exchanger against a closed loop cooling water system.

Gas from the WSA condenser, substantially free of SO_2 and sulphuric acid (barring a ppm-amount of acid mist) is further cooled after which it passes through a mist filter. Acid dropout in the mist filter is pumped intermittently to the combustor. Clean gas from the mist filter is passed to the stack by a blower. Combustion air is supplied by a blower that takes suction from the hot cooling air exiting the WSA condenser. The balance of hot air from the WSA condenser is routed to the stack for draft by means of temperature control, a slipstream is recycled through the cooling air blower to maintain a minimum cooling

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PHOTO: FLUOR

Fig. 3: Transport of converter module with SPMT.

air temperature to the WSA condenser, with any excess vented to atmosphere.

In comparison with many other WSA plants, the acid plant is more extensive featuring DeNOx and an acid mist filter. Both provisions ensure a high environmental performance (low NOx and practically no acid mist emission).

Preparation of the licensor's process design by Haldor Topsoe and Fluor's front end engineering design (FEED) packages was followed by detailed engineering, procurement and construction also performed by Fluor from its Farnborough offices in the UK.

Construction

On site preparation work started prior to a scheduled shutdown of the sour gas processing train. During the shutdown, tie-ins were installed so that the existing acid plant could continue to operate whilst the new acid plant was being constructed and then isolated once the new acid plant had started up, eliminating interruptions to production.

Fortuitously, an empty plot was available on the site for construction of the new acid plant.

A philosophy of maximising off-site construction was followed using modularisation to a significant extent. Civil/structural sections were constructed in fabrication yards, then shipped to local docks and trucked to site. The largest section of the plant that contains the SO_2 converter was shipped in sections to a temporary construction facility at the site, then the SO_2 converter vessel was installed in this module, and the module was transported to its final location using a self-propelled modular transporter (SPMT); see Fig. 3. Other parts of the plant, where modularisation made less sense, were stick built.

(Pre)-commissioning

(Pre)-commissioning activities were started in parallel with the latter phases of construction. Pre-commissioning included the usual blowing and flushing, boil-out of the steam system, firing on fuel gas, refractory dry-out (both in the combustor and in the WSA condenser), and catalyst loading (performed by a specialised contractor).

Start-up and troubleshooting

Upon completion of construction and pre-commissioning, the actual start-up of the acid plant on fuel gas was initiated after inventorying relevant systems e.g. the product acid system with concentrated acid, the boiler water system and the acid mist filter with demin water. Much of plant operation was proven on fuel gas, as this operation mode requires most equipment to operate. This operation was to be followed by normal operation on acid gas.

A number of issues within the acid plant, and their resolution, are described in the following sections.

NH_3 lance/pumps

One issue encountered was blocking of the NH_3 injection lance through which aqueous NH_3 solution is injected into the process gas for de-NOx purposes. The spray nozzle at the end of the lance features a very small opening and despite best efforts in terms of blowing lines, a blockage occurred requiring a shutdown during which the lance was pulled and cleaned. In order to prevent re-occurrence, a small duplex filtration system was fitted locally to the lance. This modification could be made very rapidly given the very small flow rate, which allowed the duplex filtration system to be made out of tubing.

This resolved the issue barring one more blockage caused by a shard of stainless steel material, almost certainly from the construction of the tubing itself.

Another issue around the de-NOx system was a mechanical problem with the reciprocating-type NH_3 injection pumps. Sticky valves on the reciprocating pump cylinder (small ceramic balls) prevented the pumps from developing the required discharge pressure. This problem was fixed by the pump vendor who replaced the original ceramic balls with stainless steel balls.

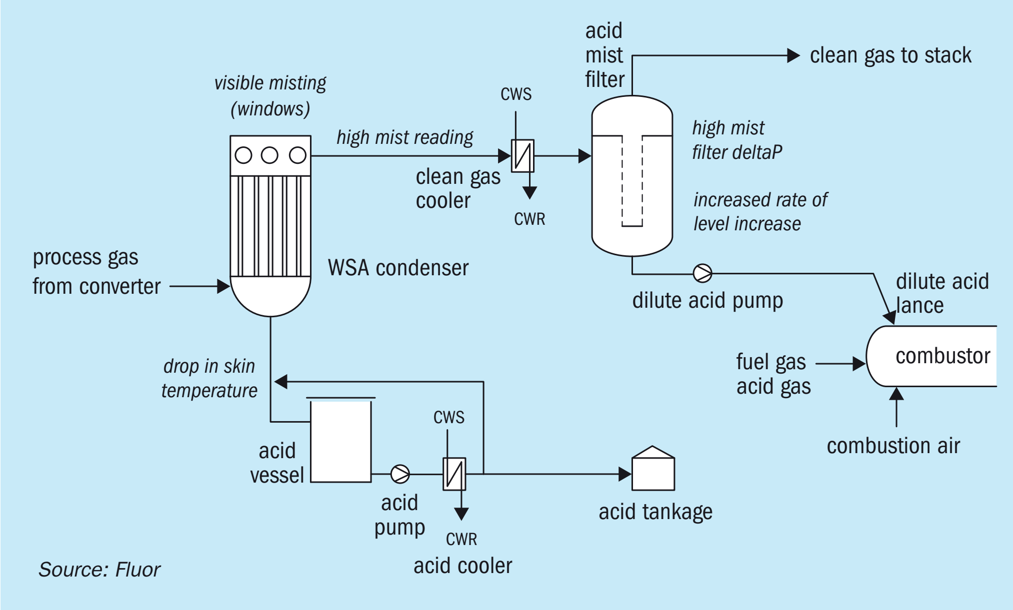
With the lance blockage and pump issues fixed, performance of the de-NOx was excellent and NOx emissions are well within spec during normal operation. Only during initial start-up, when the catalyst beds are still being heated up (and NH_3 injection is not allowed, to prevent any risk of ammonium salt deposits), NOx emissions exceed the limit for a very short time; on a time-average basis NOx emissions are very comfortably within specification.

Mist filter

A number of issues occurred around the acid mist filter system.

Initially, fairly rapid fouling occurred in the strainers in the dilute acid pump suction and discharge (the dilute acid pump moves acidic condensate entering the mist filter to the combustor). Some

Fig. 4: **Acid misting**



Source: Fluor

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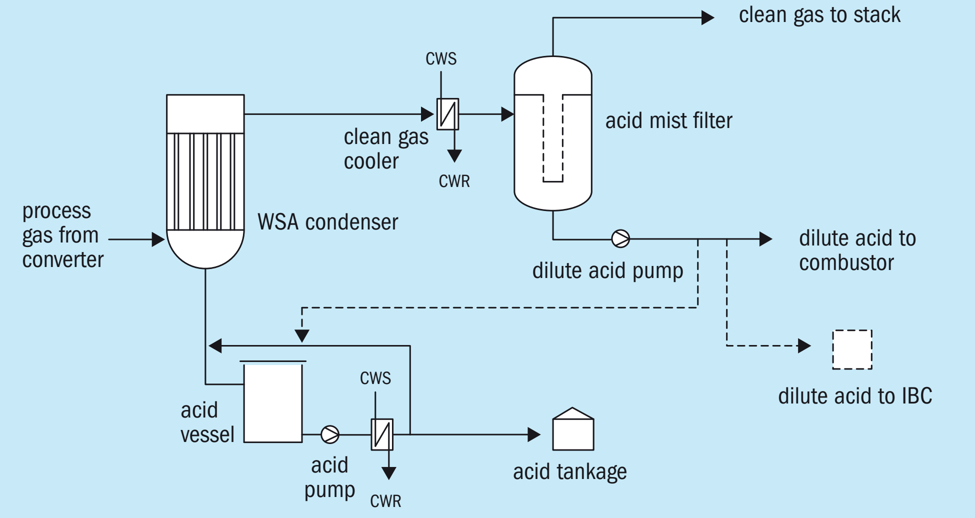
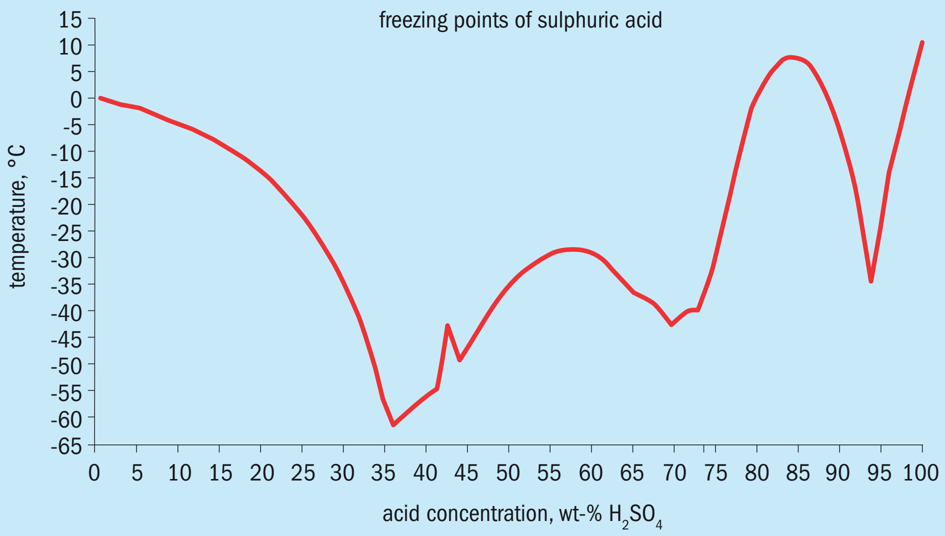
Fig. 5: **Dilute acid system**



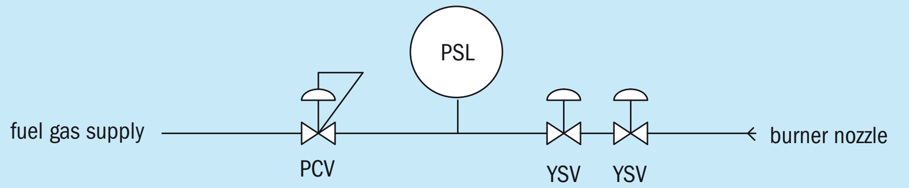
Source: Fluor

Fig. 6: **Acid freezing curve**



Source: Dow Chemical, Handbook for sulphuric acid piping systems

Fig. 7: **Pilot burner system**



Source: Fluor

fibrous material emanating from the filter candles was found (loose fibres from the manufacturing process), as well as some other material that could not be easily identified. Since cleaning the strainers required a high level of personal protection (acid resistant suits) this was a concern. The filter candles were checked for breakages, and no problems were found. Furthermore, the filter candles were rinsed in-situ with demin water during an opportunity outage. As hoped for, this issue disappeared over time, and back-up solutions developed (i.e. more easily cleanable strainers) were not required.

Another issue was a much higher than expected rate of accumulation of liquid (dilute acid) in the mist filter vessel. This was true both for fuel gas operation, and combined acid gas and fuel gas operation. Actual liquid ingress was up to 100 times higher than expected from the design material balances, even though the temperature of the process gas into the mist filter was well above the calculated water dew point and acid mist entrainment from the WSA condenser was nil or minimal. Initially, during fuel gas only operation, the high liquid ingress rate was not much of a concern, as the liquid was pumped intermittently to the combustor without any problems.

However, with the plant in combined acid gas and fuel gas operation, injection of dilute acid into the combustor resulted in an unexpected phenomenon (see Fig. 4). As soon as dilute acid injection started, a sudden and large increase in mist from the WSA condenser was observed (mist reading off-scale). At the same time, pressure drop across the mist filter elements doubled at constant gas flow (likely due to very high liquid loading). Also, the skin temperature on the acid drain line from the WSA condenser to the acid vessel dropped dramatically (indicating that hot acid was not running down but rather carried over into the mist filter). Most worrying of all, while pumping out dilute acid from the mist filter to the combustor, the mist filter level did not decrease, but kept increasing at an even higher rate than before. Eventually, after repeated attempts to stop and re-start dilute acid combustion, all with the exact same result, this situation became unsustainable and the plant was shut down in a controlled manner to avoid overfilling the mist filter (the result of which would have been dilute acid overflowing into the containment bund).

Investigation of this problem led to the following conclusions:

- Liquid ingress into the mist filter was high despite the gas temperature being above water dew point.
- Upon starting dilute acid injection to the combustor, massive misting in the WSA condenser resulted in carryover of acid that should drop out into the acid vessel into the mist filter (causing additional level rise).

The first phenomenon, the high rate of level increase in general, was attributed to localised condensation on the tubes of the clean gas cooler, with the liquid droplets not having the time to re-evaporate before impinging on the mist filter elements, even though the bulk gas temperature would indicate a dry gas. The high localised condensation hypothesis was supported by a very low

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cooling water temperature (10°C relative to the 33°C design value) as the closed cooling water system temperature control was not yet fully commissioned. When the cooling water supply temperature control (using variable speed fans on the closed cooling water air cooler) was rectified, and cooling water supply temperature was close to 33°C, a dramatic reduction in the rate of level increase was observed after re-start (from some 1.0%/hour level rise to 0.1%/hour level rise, at constant bulk gas temperature), proving the above hypothesis.

As to an explanation for the second problem, observed heavy misting as soon as dilute acid combustion started, two main explanations were offered.

One explanation was generation of a large amount of solid particles in the gas to the WSA condenser, due to various contaminants in the dilute acid, for example fibres from the mist filter candles. This could have resulted in an excessive particulate concentration in the process gas to the WSA condenser, leading to high mist formation.

The second explanation was a blockage of the atomising air nozzles in the dilute acid lance, which could result in incomplete combustion of dilute acid. The blockage was found from atomising air pressure data (higher than calculated atomising air back pressure).

The above possible causes were addressed by a thorough cleaning of the dilute acid system, requiring opening of the mist filter, the contents having been emptied into 1-m^3 plastic containers (intermediate bulk containers or IBCs), and by pulling the dilute acid lance, and removing the debris from the atomising air side of the lance and annulus of the nozzle.

Prior to re-starting, temporary alternative provisions were put in place to enable an alternative route of dilute acid disposal into IBCs through an acid resistant hose (Fig. 5).

Upon re-start, dilute acid was emptied into IBCs for a while, with full IBCs exported from site. The number of IBCs required was dramatically reduced by the above-described reduction in liquid ingress into the mist filter due to cooling water temperature control. Eventually the dilute acid combustion was re-tried. Interestingly, it was now found that the dilute acid injection actually reduced mist formation in the WSA condenser; this unexpected and favourable reduction was significant, reversible (stopping dilute acid injection increased mist again) and reproducible. With the design working again as intended, the route to IBCs was no longer required.

In order to have further flexibility, it was decided to add a provision to enable disposing of dilute acid into the acid export stream.

Needless to say such dilute acid disposal can have an impact on product acid quality with respect to strength, depending on respective flow rates of product acid (approx. 96 wt-%) and dilute acid (approx. 20-30 wt-%), and on product acid contaminants, particularly nitrogen containing compounds.

The limit on blending is that acid rundown strength should be > 91 wt-% approximately, so as to ensure a freezing point of the product acid less than the minimum ambient temperature of -10°C, as much of the export line is untraced (Fig. 6).

A temporary blending connection was made by means of an acid-resistant hose which was used for trialling blending. The main concern during trialling was the thermal effect of adding dilute acid into concentrated acid (enthalpy of dilution). The trials worked well, with very manageable acid temperature rise, at dilute acid blending rates more than sufficient to maintain stable mist filter level. A hard-piped blending connection will be added at a later time as a 'back-up' provision.

Pilot burner PCV

A fairly minor but irritating issue was experienced on the combustor pilot burner pressure regulator. Upon start of the pilot burner, the regulator could not maintain its downstream pressure. When this pressure dropped too much, the pilot burner would trip on low-low pressure (see Fig. 7).

This problem was permanently solved with a replacement of the pressure control valve (PCV).

Clean gas blower

The clean gas blower (CGB) takes suction from the mist filter and pushes the gas into the stack. The plant suffered a number of trips (thermistor) on the CGB motor. These trips only occurred after some 45-60 minutes of running the blower at maximum speed (caused by high gas flow, e.g. during combustor purge upon start-up or when operators increased combustion air to high values). What did not make sense is that the flow rate through the blower was well within the blower's curve. With electrical data-logging equipment on line, a further trip was intentionally generated by ramping up the combustion air flow, showing that the kW, Ampere and torque were all within the motor and VSD system envelope. As such, the most likely explanation is a problem inside the motor. A new motor was purchased to be fitted on an opportunity basis. In the meantime, operations limited the combustion air flow (which sets the CGB suction flow) to about 75% of design which is fully sufficient to process the amount of acid gas available until the next shutdown. No further trips were experienced.

Stable operation

The above troubles were all resolved or managed. The concerted efforts of the integrated team (owner operators, Fluor, Haldor Topsoe and suppliers) were well rewarded with stable operation. The first weeks of the start-up had only limited periods of acid gas processing (minuscule level increases in the product acid export storage tank), for reasons both inside and outside the new acid plant. Needless to say this put considerable pressure on the team. However, with the teething troubles resolved, operation was steady and successful.

With stable operation, the generally very good performance of the plant already observed during the short periods of operation in the first weeks, did in fact materialise longer term.

The main parameter of the plant is sulphur recovery (conversion of SO_2 into sulphuric acid) which was consistently above the 99.5% specification, resulting in SO_2 stack levels far below the 400 mg/Nm^3 permit limit.

Product acid quality was always comfortably within the guarantee value of 96 wt-% ± 2 wt-% .

Once issues with lance blockage and the NH_3 pumps were fixed, DeNOx performance was excellent with NOx levels below 10 mg/Nm^3 (permit 50 mg/Nm^3).

Both for CO and H_2S, combustion residuals were low; CO around 2.5 mg/Nm^3, insignificant compared to the permit limit of 100 mg/Nm^3 and H_2S 3-4 mg/Nm^3 from CEMS, relative to 5 mg/Nm^3 permit limit. The latter value from the CEMS was proven to be very conservative by manual sampling from the stack followed by lab analysis, which gave values close to the limit of detection.

The above was confirmed during the acceptance test run held at a few weeks after stable operation was established.

Acknowledgment

The author greatfully acknowledges Haldor Topsoe for the provision of Fig. 2 and wishes to thank all individuals involved in the project for their contributions.

A sustainable approach to tail gas catalysts

EcoMax™ tail gas catalysts offer a sustainable and cost effective choice for tail gas catalysts. They are made from spent hydroprocessing catalysts, which reduces the environmental and socioeconomic impacts of sourcing fresh cobalt and molybdenum and therefore supports operators' sustainability goals. **B. Visioli** of Porocel (a part of Evonik) discusses the benefits of catalyst reuse from hydroprocessing for tail gas treating.

Sustainability is not an empty buzzword in the oil and energy industries today. Corporate environmental initiatives including sustainable practices and concepts like circular economy are not only pursued for image reasons – they allow for real financial benefits and reduce impact on the environment. One way that sustainability is incorporated into the operating strategy of many refineries is catalyst reuse. In fact, more than 30,000 tonnes of regenerated and rejuvenated hydroprocessing catalysts are installed each year in hydrotreating applications. But catalyst sustainability need not be confined to the conventional hydroprocessing units.

From hydroprocessing to tail gas treating

Tail gas treating catalysts and hydroprocessing catalysts are similar in several ways. Both types of catalyst:

- are composed of one or more Group VIII metals (e.g., cobalt, nickel) and one or more Group VIB metals (e.g., molybdenum, tungsten) supported on a carrier material (e.g., alumina, silica, zeolite);
- require conversion to a metal-sulphide state, referred to as pre-sulphiding, to enable activity toward the desired reactions;
- consume hydrogen as a reactant in the desired reactions;
- are available in extruded shapes (e.g., trilobe), although spherical tail gas treating catalysts have been introduced to provide minimal pressure drop.

While there are many similarities, there are also important differences between tail gas treating catalysts and hydroprocessing catalysts.

The quantity of metal (cobalt, molybdenum, etc.) present in hydroprocessing catalysts is commonly greater than that present in tail gas treating catalysts.

In operation, hydroprocessing catalysts are not exposed to species such as SO_2, whereas this is more common in tail gas treating.

The operating pressure is significantly higher in hydroprocessing (up to 2000+ psig/130+ bar) than it is in tail gas treating.

Catalyst reuse enabled by regeneration and rejuvenation

Porocel's regeneration and rejuvenation technologies help refiners to best leverage the value of their spent catalysts, do not require significant disposal to landfill, and give the active metal components in such a hydroprocessing catalyst a second life[1]. In this way, catalyst reuse decreases the refining industry's dependence on freshly mined metals and preserves finite resources at the same time.

Regeneration converts catalytic metals to their oxide form and recovers as much activity as possible while minimising damage to the catalyst particles themselves. Regeneration processes often recover activity up to 85% of fresh catalyst activity in the same application. Regenerated hydroprocessing catalysts are typically cascaded down to a lower severity unit in place of fresh catalyst.

Porocel's Excel® rejuvenation is superior to simple regeneration. It improves dispersion of active metal sites on the catalyst by means of a chelating agent, thereby reversing the impact of metals agglomeration. Rejuvenation yields a catalyst which performs very similarly to fresh catalyst in the same application. In some cases, rejuvenation can provide even higher performance than the original fresh catalyst. Because of the higher degree of recovered activity recovery, a rejuvenated catalyst may be loaded into the same application it was harvested from or one of similar severity.

Fig. 1. Shows a schematic of refinery catalyst reuse including EcoMax™.

Porocel has decades of experience with these catalyst recovery processes and hydroprocessing applications, which is a critical component in the catalyst reactivation and reuse system. Porocel's expertise ensures that catalyst with the right level of activity and selectivity is properly applied, whether a refiner uses catalysts from their own catalyst reuse pool or purchases the catalyst from Porocel's inventory of high-quality regenerated and rejuvenated hydroprocessing catalysts.

Rethinking the source of tail gas hydrogenation catalyst

Tail gas treating catalysts have been available since the 1980s, but there is still room for improvement in this established technology. Porocel's EcoMax™ catalyst technology leverages decades of catalyst reactivation and reuse experience to extend its benefits

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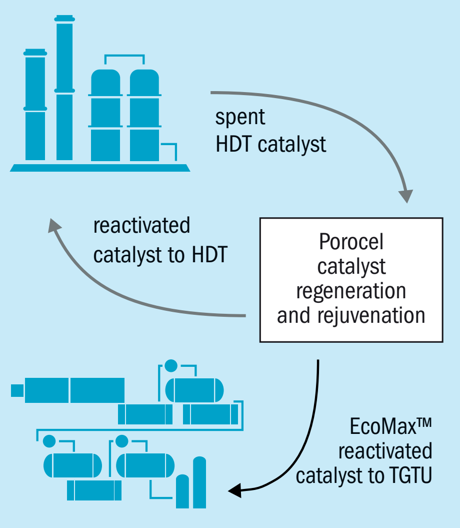
Fig. 1: **Schematic of refinery catalyst reuse including EcoMax™**



Source: Porocel

to the tail gas treating unit: it treats a spent hydroprocessing catalyst in a catalyst reactivation facility to remove contaminants and to optimise the catalyst for use in a tail gas treating unit's hydrogenation reactor.

Maximise the retained value of catalyst

When a refiner purchases a hydroprocessing catalyst, the refiner has invested in an asset composed of the materials in the catalyst itself, as well as the technology and research which went into developing that catalyst. The value of this asset does not disappear completely over its active life, nor does it suddenly dissipate at end of run. Typical catalyst disposal methods not only add processing cost, they also fail to leverage the catalyst investment in the best way possible.

Catalyst reactivation and reuse not only reduces the cost associated with catalyst disposal but can even result in a reimbursement to the refiner. Porocel provides a credit to refiners in return for acquisition of their spent catalysts to value the catalysts' future reuse. EcoMax™ paves the way for new catalyst reactivation and reuse designs.

Reduce expenditure of replacement catalyst

Sulphur recovery facilities operate primarily as a means of environmental control, rather than a strong profit centre, despite elemental sulphur being a critical commodity in today's technological landscape[2]. As such, expenditures related to sulphur recovery are under scrutiny, extending to selection and procurement of catalyst. One obvious benefit of EcoMax™ technology is that it is less expensive than catalyst made from fresh materials. The primary driver for this value improvement is that procurement or production of fresh catalyst substrate and active metals components can be avoided. EcoMax™ catalyst typically yields a 30-50% cost reduction versus fresh catalyst, in addition to the other benefits mentioned here.

Reduce SRU emissions by improving catalyst activity

The sulphur recovery unit (SRU) is first and foremost an emissions control unit. A catalyst with high activity will maximise its efficiency and minimise emissions. In particular, the conversion efficiency of COS and CS$_2$ in the tail gas reactor will have a direct impact on SO$_2$ emissions from the sulphur plant incinerator or thermal oxidiser stack. EcoMax™ catalyst performs at least as well as benchmark catalysts at both conventional temperature and low temperature conditions, especially in conversion of these carbon-sulphur compounds.

This is shown clearly in Fig. 2, where conversion achieved with EcoMax™ is substantially above the benchmark range for COS and CS$_2$. In these experiments, EcoMax™ catalyst was tested at both conventional temperature (280°C/535°F isothermal bed temperature) and low temperature (240°C/465°F isothermal bed temperature) conditions. This corresponds to reactor inlet temperatures of approximately 250°C and 220°C, respectively.

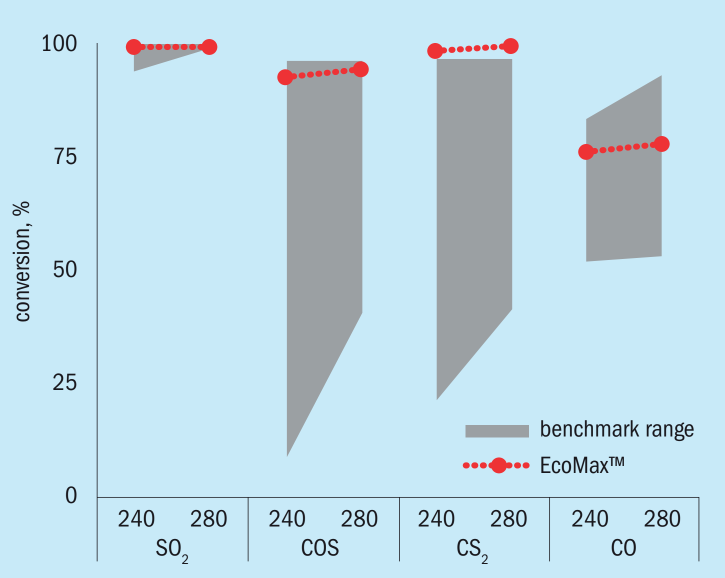
Reduce energy consumption for reactor feed heating

The tail gas treating unit configurations which are most often employed in the oil and gas industry consume a significant amount of energy to reduce emissions by a small percentage[3]. The primary benefit of high-activity catalysts is that they may be operated at low temperature to reduce energy requirements. As demonstrated above, EcoMax™ catalysts display similar or greater activity than benchmark catalysts, especially for COS and CS$_2$ conversion, even at low temperature conditions. With this increase in activity EcoMax™ catalyst enables the operator to reduce energy consumption and CO$_2$ emissions from operations at the same time.

Decrease negative environmental and social impacts

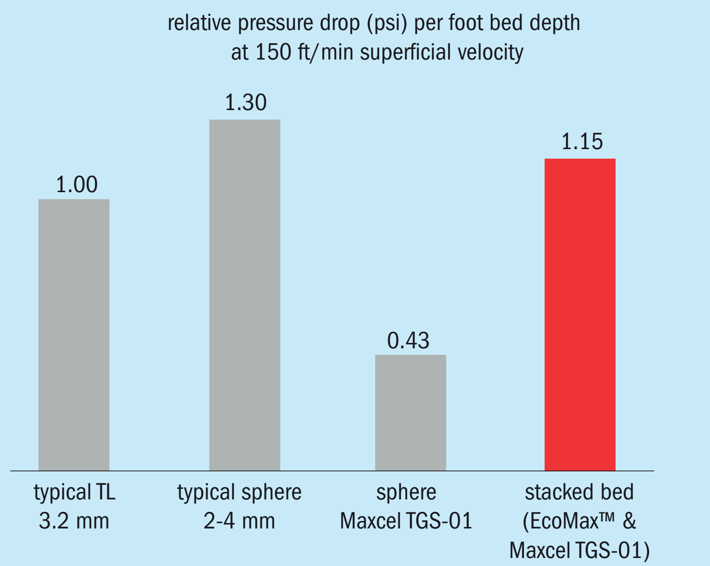
The oil and gas industry must ensure that investment and operating decisions are made with environmental considerations. Tail gas

Fig. 2: **High performance of EcoMax™ catalysts**



Source: Porocel

Fig. 3: **Pressure drop characteristics of common TG catalysts**



Source: Porocel

catalysts made from fresh materials result in the consumption of a surprising amount of energy: nearly 15,000 pounds of CO_2 emitted (CO_2e) per tonne of catalyst produced! Cobalt mining has also come under criticism for inhumane working conditions. “Artisanal mining” in certain regions (estimated to be responsible for more than 10% of the world’s copper-cobalt ore output[4]) exposes workers to a hazardous working environment over long working hours, with little pay[5,6,7].

To manufacture fresh tail gas catalyst, raw materials must be sourced (ultimately from mines), converted into catalyst feedstocks[8,5], and formed into a catalyst. This process is energy intensive and has significant environmental consequences. However, when a catalyst can be reused by the EcoMax™ process, more than 70% of the carbon emissions associated with these manufacturing processes (tens to hundreds of tonnes of CO_2e per reactor) are eliminated.

Case study for a pressure drop-constrained unit

Some sulphur recovery units are constrained by equipment pressure drop. The feed gases to most SRUs are provided only slightly above atmospheric pressure, and this means that a small increase of one psi (a few kPa) of pressure drop can make the crucial difference between normal operation and a unit bottleneck. To prevent these issues, some facilities elect to use a spherical tail gas catalyst with lower pressure drop. Given that EcoMax™ catalysts are extruded, can the cost savings of EcoMax catalyst be realised for a unit constrained by pressure drop?

Fig. 3 shows the measured pressure drop at 150 ft/min superficial velocity over two benchmark catalysts as well as Maxcel TGS-01. All else equal, EcoMax™ catalysts will show greater pressure drop per unit bed depth due to their smaller particle size, especially compared to the low-pressure drop tail gas catalyst, Maxcel TGS-01. However, a stacked bed approach may be used to achieve the cost savings of EcoMax™ without sacrificing pressure drop.

In this example, a configuration of 85% EcoMax catalyst plus 15% Maxcel TGS-01 yields a pressure drop within the range of the benchmark catalysts. It is estimated that even with the supplement of Maxcel TGS-01 fresh catalyst, a cost savings of 20-40% can still be achieved. ■

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New low temperature TGT catalyst from Süd-Chemie

HDMax® 230 is the 3rd generation low-temperature CoMo-type hydrogenation catalyst from Süd-Chemie (a Clariant Group Company), specially designed to ensure long and trouble-free operation of tail gas treatment (TGT) units. HDMax® 230 hydrogenates all sulphur species at low temperature, including SO_2, CS_2, COS and Sx, while CO is converted to CO_2 via the water gas shift reaction and can be applied to most sulphur removal technologies.

One of the most unique attributes of HDMax® 230 is its ability to attain the required activity with significantly lower amounts of metals compared to most other catalysts in the industry for the same application, thus providing high performance benefits with minimal utilisation of natural resources.

HDMax® 230 can also be used as a drop-in in conventional high temperature applications and benefit from significant energy savings.

In accelerated aging tests catalyst was exposed to a steam-to-gas ratio of 1.0 at elevated temperature of 500°C for hydro-thermal aging for three cycles. In actual operation, catalyst is unlikely to be exposed to such extreme conditions, however, for developmental purposes extreme conditions were considered to ensure that the catalyst is able to withstand any unforeseen process upsets. The COS conversion achieved was >95% for aged HDMax® 230 compared to 97% for fresh catalyst.

In pilot tests for CO conversion via the water gas shift reaction HDMax® 230 achieved an outlet analysis of 0.048 (wet mol-%) or 0.072 (dry mol-%), which were close to CO equilibrium.

HDMax® 230 catalyst sulphiding at lab scale is carried out at a low temperature of 240°C, since TGTUs designed for low temperature are unlikely to sulphide catalyst at temperatures higher than 280-300°C. The performance evaluation was as expected with a % COS conversion of approximately 97%. There are a number of operating plants where the second stage sulphiding was carried out at 227°C and 233°C and they are performing satisfactorily.

The unique formulation of HDMax 230® provide several performance and commercial benefits:

- enhanced activity due to a proprietary doped promoter at low temperature of 220°C;
- optimum metals loading ensuring very high surface dispersion;
- stabilised carrier to resist thermal aging to prolong catalyst life;
- custom tri-axial shape to maximise geometric surface area and lower pressure drop;
- outstanding physical strength to withstand operational upsets;
- reduced preheat temperatures for energy optimisation.

To date, HDMax® 230 has been operating in multiple units for over five years. New charges of HDMax® 230 are likely to start up by mid-2021 at Indian Oil Corporation Limited (IOCL), Hindustan Petroleum Corporation Limited (HPCL), Bharat Petroleum Corporation Limited BPCL) and Mangalore Refinery and Petroleum Ltd (MRPL). HDMax® 230 is currently operating in SRUs licensed by Black & Veatch, Engineers India Limited and KT – Kinetics Technology, and by mid-2021, in many other technologies. ■

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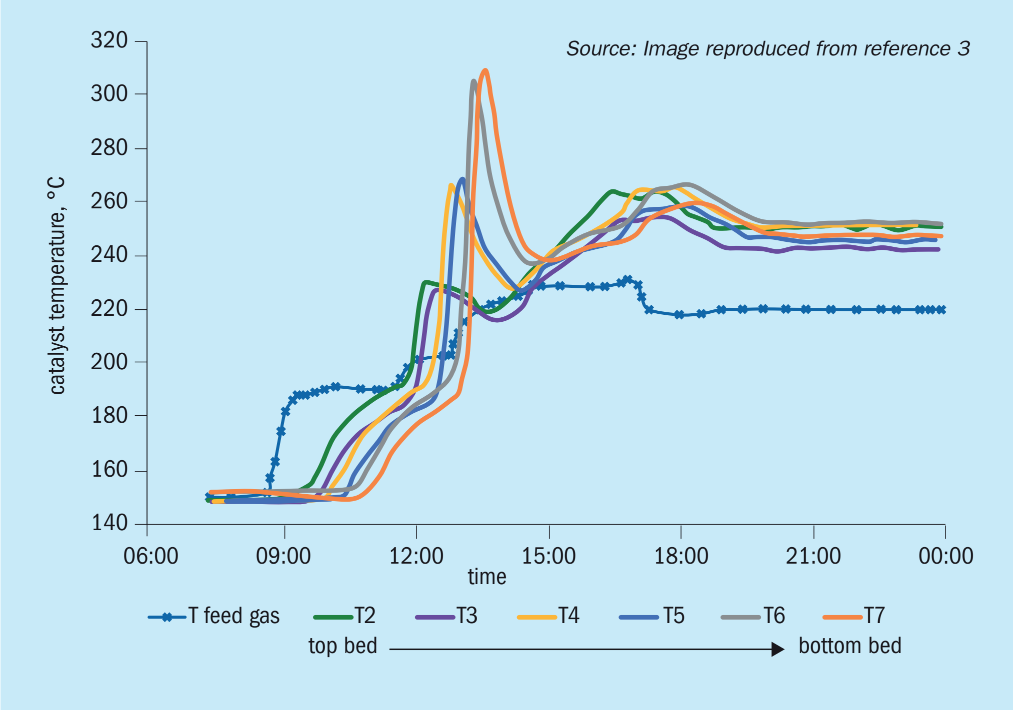
The ease of titania-based tail gas catalysts start-up

Euro Support's new generation, titania-based low temperature tail gas catalyst shows a marked robustness to the start-up procedure compared to its common alumina-based counterparts. **Dr B. Hereijgers** of Euro Support discusses the promising lab results.

In *Sulphur* no. 369 Euro Support introduced its titania supported tail gas catalyst and highlighted the operational benefits on offer, especially for TGTU installations operated with steam reheaters at low temperature[1]. Since then, Euro Support has received increasing signals from the field of users of low temperature tail gas treating units (LT-TGTU) loaded with traditional alumina-based LT catalysts that in-service operations are often fraught with hiccups. In particular, serious challenges exist to maintain the required low emissions over time.

When considering the operation of an LT-TGTU installation special attention to the catalyst start-up procedure is justified. As recently presented by Sulphur Experts, this involves a time-consuming and complex procedure whereby pristine catalyst is exposed to hydrogen and hydrogen sulphide containing gas to be transformed into the active sulphide phase[2]. It has been shown multiple times in literature that a successful sulphidation is a measure of strict temperature control. For the high temperature application, the exothermic reaction should be curbed to prevent sintering of the catalyst, whilst under low temperature start-up conditions the main challenge is to make sure the exothermic reaction can heat up a sufficiently large portion of the catalyst bed to allow for deep sulphidation of the metals. Either way, the procedure requires tight process control and specific gas feed conditions, meaning that in the meantime the tail gas is routed to the incinerator leading to increased emissions. The big advantage of a high temperature system here is that a failed sulphidation can be restored by heating the bed in H_2S/H_2 to a temperature above 300°C. In case of a low temperature system the inlet temperature is simply limited to 240°C and once the catalyst is partially sulphided, an exothermic reaction is circumvented.

Fig. 1: **Claus 4 SCOT DC 1903 temperature profile during in-situ sulphidation**

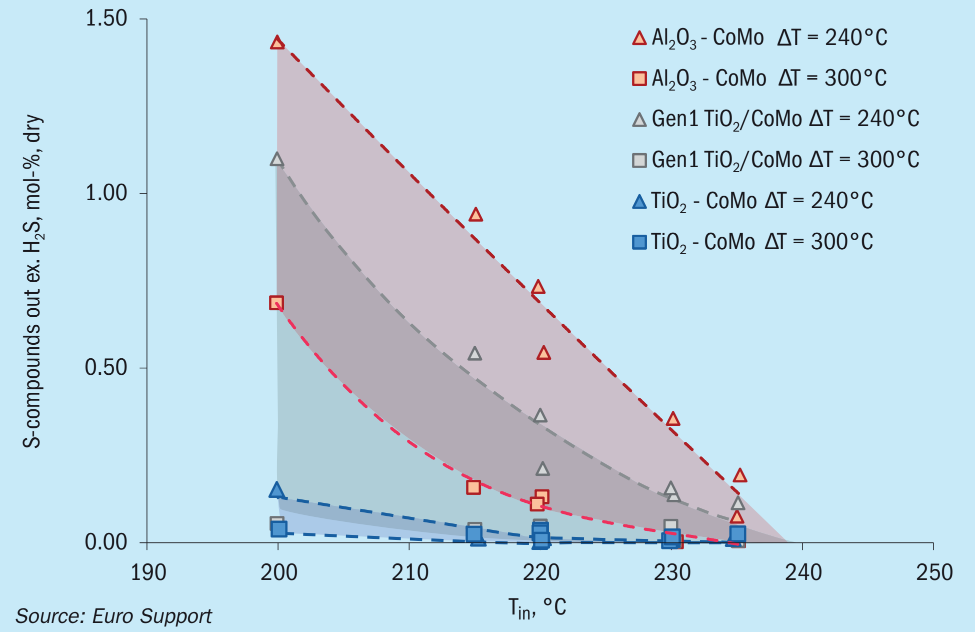


Source: Image reproduced from reference 3

In a publication by Crevier *et. al.*[3] from 2009, the actual temperature wave in an industrial reactor loaded with low temperature tail gas catalyst TG-107 was monitored in detail. A reproduction of the corresponding data is presented in Fig. 1. The conclusion was that about one third of the catalyst bed can reach a temperature above 300°C for a short time, whilst the remaining two thirds reached a maximum temperature around 260°C which could be sustained for 1-2 hours. This means that in practice, even when a perfect start-up procedure is performed, maximum one third of the loaded catalyst will be reasonably sulphided. Huffmaster *et. al.*[4] reported that such a catalyst sulphided at 260°C can reach between 66-83% of the activity compared to the activity reached after start-up at 300°C and 10% hydrogen concentration. It should be stated though that even sulphidation of an alumina-based tail gas catalyst at 300°C is not as effective as a real high temperature sulphidation[1]. In reality, the reduced activity that can be expected may cause a slip of sulphur compounds through the reactor. The nature of the different compounds can cause different operational problems for the TGTU as a whole; from increased SO_2 emissions by COS and CS_2 slip to the incinerator to sulphur fouling of the quench water and increased amine degradation by S_8 and SO_2 slip.

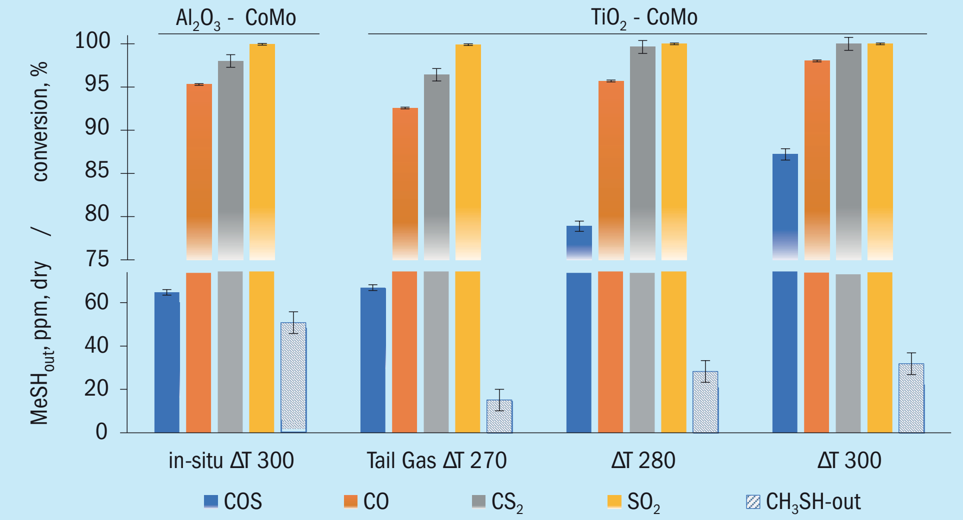
In this article Euro Support presents a new generation titania-based tail gas catalyst and shows the impact it has on the ease and robustness of the in-situ start-up procedure compared to its traditional alumina-based counterparts.

Fig. 2: **Total sulphur compounds in the reactor outlet as function of inlet temp.**



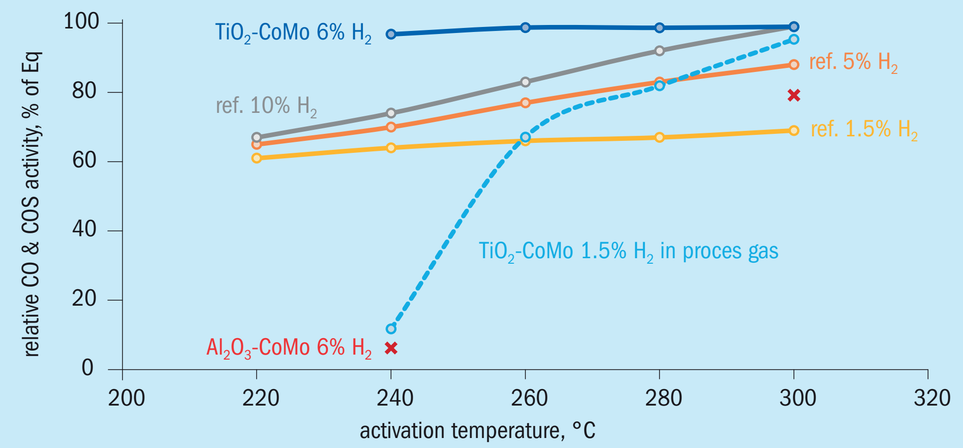
Source: Euro Support

Fig. 3: **Catalyst performance at T_{in} = 240°C of TiO_2-CoMo after exposure to the indicated maximum temperature**



Source: Euro Support

Fig. 4: **Averaged CO and COS conversion activity during start-up**



Source: Euro Support

Results and discussion

Fig. 2 displays the total concentration of sulphur compounds (SO_2, CS_2, COS, methyl mercaptan and elemental sulphur, excluding H_2S) found in the reactor outlet of the TGTU catalyst as function of the reactor inlet temperature and maximum reached in-situ sulphidation temperature. This represents the total sulphur load that was not converted to H_2S. The largest absolute contribution to the slip is in the form of elemental sulphur. The concentration of sulphur compounds was measured dry and free of elemental sulphur. The total sulphur load in the input gas was 2.0 mol-%.

In the graph, the performance of Euro Support's new titania-based catalyst is compared to the alumina-based LT-TGTU catalyst and the 1st generation TiO_2/CoMo TGTU catalyst. Performance is plotted for two conditions where ΔT 240°C and ΔT 300°C indicate the maximum temperature that is reached by the heat wave from the exothermic reaction during in-situ sulphidation. These represent 'worst-case' (triangle) and 'perfect' (square) start-up procedures, respectively. The shaded area in between these lines (red for alumina and blue and grey for titania) represents the activity that can realistically be expected from the LT-TGTU catalyst. The graph for the alumina-based LT-TGTU catalysts shows that after perfect in-situ sulphidation an operation temperature of above 230°C is required to prevent slip of sulphur compounds already at start of run. The data suggests that a worst-case start-up makes it virtually impossible to operate at full conversion. In practice, the activity will fall in between in the shaded area, but in any event leaves limited room for the inevitable catalyst deactivation over time.

Although the first generation of TiO_2-CoMo catalyst readily outperforms an alumina-based catalyst under similar sulphidation conditions, the possibility to operate at start of run in the T_{in} < 230°C range still depends largely on the success of the sulphidation treatment, as indicated by the grey area in Fig. 2.

The new TiO_2-CoMo catalysts show a completely different picture. At inlet temperatures above 210°C both catalysts sulphided at 240°C or 300°C perform equally well and no slip in unconverted sulphur compounds was observed. This means that if the heat wave during in-situ start-up is lower than expected, because of not having the optimal conditions in the reactor, the catalyst will still

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be sulphided almost to its full potential, with no significant effect on catalyst activity.

From the data in Fig. 2 it can be concluded that TiO_2-CoMo shows a remarkable high activity towards sulphidation under mild conditions. This result raises the obvious question whether it would be feasible to successfully start-up a TGTU catalyst by simply exposing the pristine catalyst to Claus tail gas. After all, this would avoid the complex procedure and the chances of mishaps and permanent loss of catalyst activity.

Fig. 3 presents the performance of the TiO_2-CoMo catalysts at T_{in} = 240°C when no start-up treatment is performed. Activity is shown as a function of temperature (ΔT) that the catalyst was exposed to for four hours in the same Claus tail gas that was also used for the activity tests. The Al_2O_3-CoMo catalyst after best possible in-situ sulphidation at 300°C, is included in the figure for comparison.

It was found that when Euro Support's new generation of TiO_2-CoMo catalyst is exposed to Claus tail gas at T_{in} = 270°C for four hours its performance roughly matches that of Al_2O_3-CoMo after the best possible in-situ start-up. When the temperature was increased slightly the performance could be enhanced even further. Although more research will be performed by Euro Support in the near future, these results already indicate that it is feasible to start a fresh TiO_2-CoMo TGTU catalyst without performing a specific start-up procedure. By optimising the gas composition to the right $H_2S:SO_2$ ratio and space velocity, performance may likely be enhanced further.

Recently Shell Catalysts & Technologies published a paper on effective activation of alumina-based low temperature tail gas catalysts[4]. Although the reported sulphiding and test conditions are not identical to what are practiced at Euro Support's labs, a useful benchmarking of results is provided. Generally speaking, the start-up and test conditions in Euro Support's tests are comparable, but somewhat more stringent to the catalyst, as compared to Shell's reported stress test.

In Fig. 4 the data, as reported in the paper[4] for a "representative low temperature TGU catalysts", is compared with Euro Support's test results for titania and alumina based catalysts under comparable start-up and operating conditions. Here activity is represented by the average CO and COS conversion as a fraction of the calculated equilibrium conversion[5]. The data from Shell show a clear decrease in hydrogenation activity when sulphidation temperature or hydrogen gas concentration are decreased. Full conversion (99% of equilibrium) is only achieved after activation at 300°C in 10% H_2, which is not realistic under normal conditions. The best performing alumina-based catalyst only reached 79% of the equilibrium conversion under optimal sulphiding conditions, as applied in Euro Support's tests. Euro Support's new generation titania based CoMo catalysts compares favourably to all the alumina-based counterparts. Already at a sulphiding temperature of 240°C (meaning no exotherm in the bed), 97% of the equilibrium conversion is reached. If a minor exothermic reaction is experienced, that drives the temperature to 260°C, the catalysts reach full (99%) CO and COS conversion. Under all circumstances, the TiO_2-CoMo catalyst performs significantly better than any alumina based CoMo catalyst.

The dashed line in Fig. 4 represents the normalised CO and COS activity for this catalyst after exposing the catalyst to standard Claus tail gas with a $H_2S:SO_2$ ratio of 2, at the indicated temperature. Under these conditions the catalyst, albeit almost, cannot be sulphided to its full potential. On the other hand, considerably higher activity is still achieved compared to the alumina-based low temperature catalyst that is considered the market's standard, in the same test. When a temperature above 280°C is reached in the catalyst bed with Claus tail gas, the activity is in the range of what would be considered as acceptable. If the start-up conditions were optimised to a higher $H_2S:SO_2$ ratio and increased H_2 concentration the performance could likely be enhanced.

Conclusions

Euro Support's new generation titania-based low temperature tail gas catalyst shows a marked robustness to the start-up procedure compared to its common alumina-based counterparts. Catalyst performance tests show that the alumina-based CoMo catalysts hardly get sulphided sufficiently under best possible in-situ start-up conditions to obtain an acceptable activity where no sulphur slip into the quench tower can occur, even at start of run. By contrast, the titania-based CoMo catalyst can be sulphided effectively in a worst-case scenario where virtually no exotherm in the catalyst bed is achieved.

Euro Support's titania-based tail gas catalyst proved it is feasible that this kind of TGTU catalyst can exhibit sufficient activity even when no start-up procedure is applied. A simple activation in process gas under normal operation conditions has proven to be sufficient in the lab. More research is ongoing to map out the boundary conditions for successful operation.

Euro Support benchmarked the test data to literature performance data and although test and start-up conditions are different, the comparison underscores the superior performance of TiO_2-CoMo compared to its alumina-based counterparts under realistically applied start-up conditions. ■

Acknowledgements

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Experimental notes

All catalyst tests reported were performed in the test laboratories at Euro Support Manufacturing Czechia s.r.o. in a dedicated glass bench-scale test reactor of 30 mm I.D. loaded with 70 mL shaped catalyst.

In-situ sulphiding conditions:
H_2S = 1.5 mol-%, H_2 = 6.0 mol-%,
H_2O = 6.7 mol-%. GHSV = 450 h^{-1} STP

Claus tail gas composition:
H_2S = 1.0 mol-%, SO_2 = 0.5 mol-%,
CO = 1.1 mol-%, H_2 = 1.5 mol-%,
COS and CS_2 = 250 ppm,
CO_2 = 16.7 mol-% and H_2O = 22 mol-%.
GHSV = 1500 h^{-1} STP

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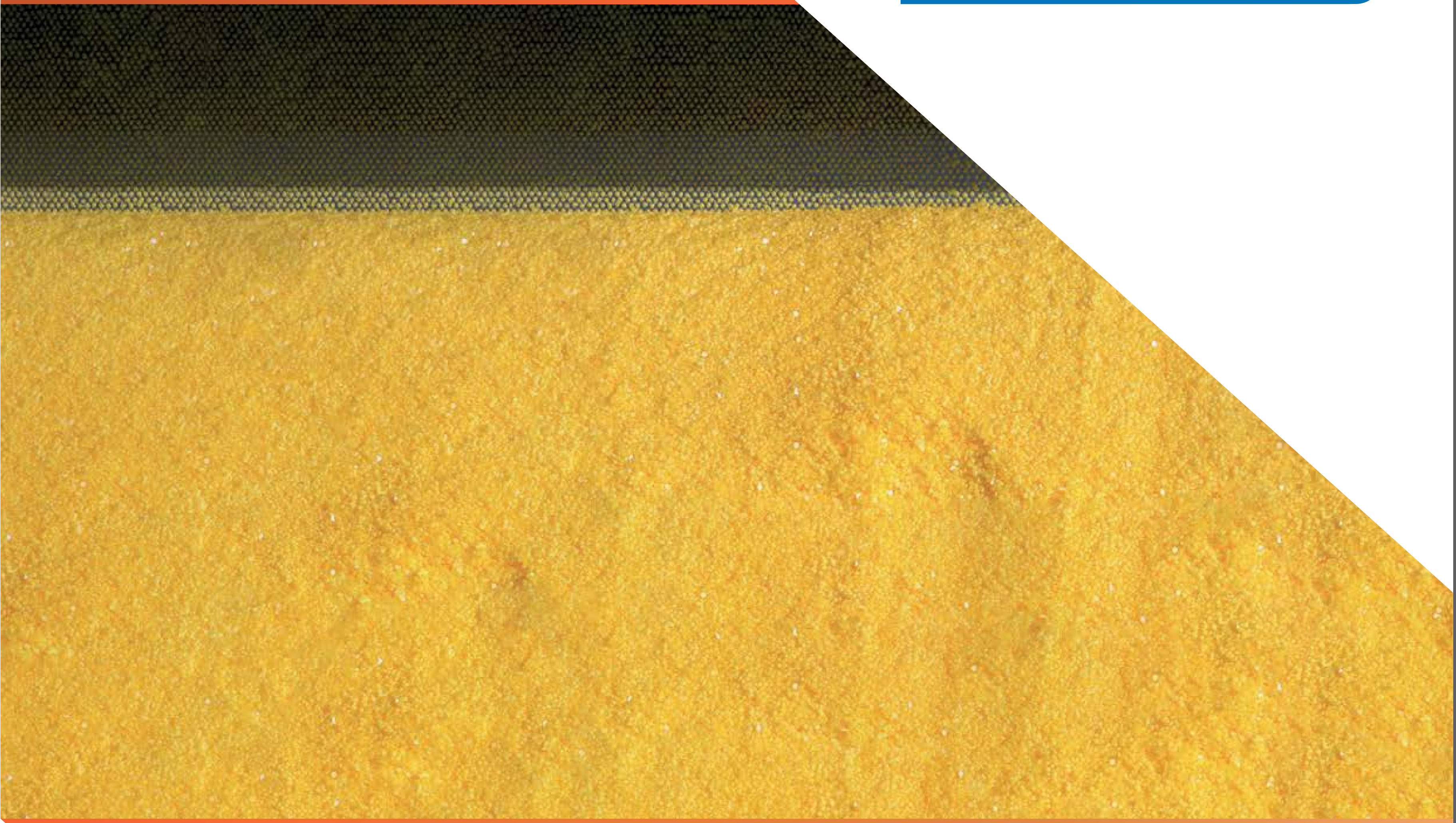

Better monitoring of sulphuric acid and oleum

Automatic identification and optimum online monitoring using sonic velocity measurements can now be used to determine the concentration of both sulphuric acid and oleum. **C. Kahrmann** and **T. Knape** of SensoTech report on a new user-friendly method of measurement providing a significant step towards automated process control.

Sulphuric acid is still one of the world's most important industrial chemicals, used in many industrial applications and production steps. It is produced by the double contact double absorption process (DCDA) or the wet gas absorption process (WSA). Exact monitoring of the concentration of sulphuric acid and oleum in sulphuric acid plants is of great importance as it determines the quality of the end product. It is precisely this determination of the concentration that can be ideally realised by means of sonic velocity.

Conventional monitoring of sulphuric acid and oleum is carried out in many production facilities by taking samples manually. These samples are then analysed in the laboratory and a result is available a few hours later.

Advantageous of sonic velocity for process control

Determining the concentration of sulphuric acid by measuring the sonic velocity, however, offers several advantages over many other measurement methods.

Measurement principle

By emitting sound waves from a sound emitter to a receiver, the transit time of the sound wave is measured several times per second. With the measured transit time and the known distance between the emitter and the receiver, the sonic velocity is precisely determined in the electronics of the sensor. Since the sonic velocity is influenced by temperature, this must also be measured in the process liquid and its influence included in the calculation of the sonic velocity. Both sulphuric acid and oleum show excellent dependencies of the temperature-compensated sonic velocity on the concentration. The concentration can be calculated from these dependencies with the aid of a calculation model in the controller and shown on the display.

Measurement of sulphuric acid concentration

Sonic velocity is ideally suited to the monitoring of sulphuric acid concentration, providing a user-friendly method of measurement, especially in the target concentration range 80-100% H_2SO_4.

Monitoring can be performed with an accuracy of up to ±0.03% and, unlike many other measurement systems, thanks to the static and dynamic temperature compensation in the LiquiSonic® system, temperature does not influence the measurement of the sonic velocity and density. It can be installed freely in the process and, compared to the density measurement method, it is less sensitive to emerging contamination in the process liquid.

Boliden confirms the suitability of sonic velocity

In Harjavalta, Boliden produces the main products copper, nickel, gold and silver, as well as by-products such as sulphuric acid. Traditionally, Boliden has used a conductivity analyser for H_2SO_4 concentration measurements. At its new sulphuric acid plant, however, Boliden wanted to test something else to see if better accuracy and reliability could be achieved using an alternative technology.

The SensoTech analyser was selected due its accuracy statement and robustness. The accuracy of the first LiquiSonic® measurement was proven according to specification (0.05 wt-%) over the whole measuring span.

"All the units installed have been working fine since commissioning. The Boliden laboratory takes a control sample once a day from each line for comparison which has confirmed the accuracy. With conductivity analysers inaccuracies led to the need for many new calibrations. The reliability of the system was not at the level the operators required. Also, the measurements are almost useless when the acid concentration goes below 94 wt-%, due to the flat concentration curve of the conductive method. LiquiSonic® is in agreement with the lab references and re-calibration is no longer needed", confirmed Jarkko Heikkilä, head of maintenance.

The first LiquiSonic® concentration analyser went into operation at the new sulphuric acid plant in Harjavalta in August 2018 to measure sulphuric acid concentration 90-100 wt-% at the outlet of the plant to the storage tank (Fig. 1).

In 2019, more units were purchased to replace conductive analysers in the old sulphuric acid plant – one at the outlet of the plant to the storage tanks, another for the feed line to the drying tower, plus one for the return line of the final absorption tower (Fig. 2).

PHOTO: SENSOTECH

Fig. 1: LiquiSonic® controller with plastic field housing.

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Oleum monitoring during production

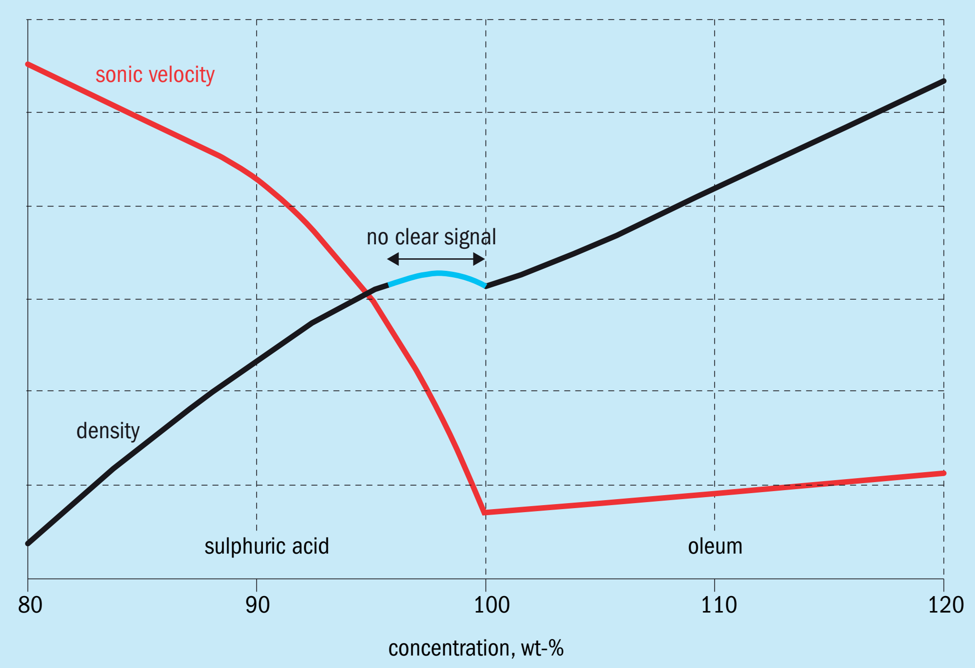
Oleum is a solution of sulphur trioxide in sulphuric acid and is formed during concentration in sulphuric acid production. As soon as the sulphuric acid reaches a concentration of 100%, any further introduction of sulphur trioxide (SO_3) is dissolved in the sulphuric acid. It is present as “free SO_3” and is displayed as such by LiquiSonic® for the oleum range.

Oleum is also called “fuming sulphuric acid”, because in pure air the dissolved sulphur trioxide in the sulphuric acid immediately escapes. Furthermore, oleum has a strongly hygroscopic character, so that even the water content in the air causes a falsification of the initial concentration. Because they are classified as hazardous chemicals, high safety precautions must also be taken when handling them. The validity of samples analysed in the laboratory is therefore critical, even with the most stringent measures in place. Moreover, taking a sample results in a time delay. Samples have to be checked in the laboratory, which means that measurement results are only available hours later. Users can therefore only react to deviations in the process at a very late stage. Inline measurement therefore is highly recommended in technical production.

PHOTO: SENSOTECH

Fig. 2: LiquiSonic® inline immersion sensor.

Fig. 3: **Dependency of the concentration and sonic velocity/density**



Source: SensoTech

The concentration range of dissolved sulphur trioxide in sulphuric acid (oleum range) can also be monitored at sonic velocity. In the range 20-35%, a clear signal is obtained with sonic velocity measurements and thus a very accurate concentration determination. Fig. 3 shows the relationship between the concentration of sulphuric acid and dissolved sulphur trioxide in oleum and the specific sonic velocity.

Since both sulphuric acid and oleum are extremely aggressive chemicals, the sensors are made of materials that are resistant to aggressive chemicals. Thus, LiquiSonic® sensors are characterised by a long service life. For application in sulphuric acid and oleum typically Hastelloy C2000 is used.

Sulphuric acid and oleum simultaneous monitoring

Oleum or sulphuric acid usually occur independently of each other during production. Until now, it was common practice to precisely configure one analyser for a correct inline measurement. To distinguish between sulphuric acid and oleum, a second physical quantity, the density, is used. In the oleum range, this exhibits a linear dependence on concentration.

The LiquiSonic® controller uses a feature specially developed for this requirement. The detection and switching between the ranges are thus fully automated. The concentrations are determined with a special calculation model, displayed and, if desired, passed on to a process control system.

Compared to the separate inline measurement of both components, the simultaneous inline measurement has clear advantages. While the respective calculation model must be selected via the controller for individual measurements, this step is completely eliminated by using a single model. This has the advantage that concentrations, especially around the switchover point, can be determined exactly at any time. If necessary, the maintenance-free LiquiSonic® measuring system offers

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Fig. 4: The LiquiSonic® analyser for sulphuric acid and oleum monitoring.

PHOTO: SENSOTECH

the possibility of adjusting the calculation model to emerging contamination, gas bubbles and other factors that interfere with the measurement. The device can thus be used very flexibly and adapted to changing process conditions.

Smart process monitoring with LiquiSonic®

Especially for the monitoring of sulphuric acid and oleum, sonic velocity sensors provide highly accurate measurements. Users are thus able to optimise their process in a simple way. Continuous monitoring and documentation of quality and productivity is the basis for lean, cost-efficient and safe processes.

The LiquiSonic® measuring system from SensoTech GmbH (Fig. 4) enables monitoring of sulphuric acid and oleum with only one measuring device and without manual switching.

Automatic product recognition

Due to the different physical properties of oleum and sulphuric acid, LiquiSonic® recognises which substance is flowing through the line. While the oleum concentration shows a linear dependence on density and sonic velocity, sulphuric acid shows a different behaviour. As the concentration increases, the sonic velocity decreases exponentially, with density having a reversal point at 98%. The calculation method developed by SensoTech uses this opposite behaviour for product detection. The intelligent controller always displays the correct concentration of the current process liquid. This smart process solution thus effectively prevents misinterpretations.

Finally, in this application, the controller also provides warning messages about detected deviations in the process, which helps to improve the safety of people and the environment. The user is always informed about the current status of the process. These messages are also recorded in the automatic documentation, which saves an enormous amount of time for users and improves subsequent error analysis.

Increase safety, save resources

The simultaneous monitoring of sulphuric acid and oleum with LiquiSonic® thus revolutionises the workflow in production plants. By eliminating time-consuming and inaccurate offline sampling with the help of simultaneous inline measurement, an increase in quality assurance can be achieved immediately after installation of the measurement system. Due to the avoidance of direct contact between people and process liquid during sampling, inline measurement ensures protection of people and the environment. Automated switching between the measured process liquids provides another safe measurement. Time-delayed or forgotten switchovers between the measuring ranges, which lead to incorrect measurement data, are thus no longer a problem. The comprehensive documentation function enables long-term storage of the measurement data and subsequent analysis of the process. The data can be read out manually at the controller or transmitted directly to the process control system. Continuous data acquisition and its transfer to the process control system helps to avoid faulty batches. This enables optimum measures to be taken to adjust the process – for example, automated re-batching or manual intervention in an emergency. In this way, a purposefully controlled process sequence is realised, which maximises plant utilisation and use of resouces.

Maintenance-free measurement

Sonic velocity sensors are robust, they contain no moving parts and are ideal for use in harsh environments. The maintenance-free and long-lasting use of the sensors result in time and cost savings for repairs that would otherwise occur.

Result

The LiquiSonic® measuring system ensures optimum monitoring of sulphuric acid and oleum. Measurements directly in the line increase process reliability. Immediate warning messages ensure extremely short reaction times in the event of process deviations, reducing the required resources and ensuring improved product yields.

In most cases, the measurement system pays for itself after one year at the latest. This has convinced numerous users, including Boliden in Harjavalta, to install the system. On the one hand, this is due to the fact that the system is maintenance-free and has a long service life. On the other hand, the time savings due to inline measurements and the optimal use of resources also come into play. Monitoring takes place with no intervention in the process. Thus, the LiquiSonic® measuring devices represent a significant step towards automated process control. The sensors automate many steps of process monitoring and reduce human error. ■

A tale of two acid tower systems

All acid towers eventually require replacement. In this article, **K. Sirikan**, **A. Mahecha-Botero** *et al* of NORAM Engineering and Constructors Ltd discuss two recently executed acid tower replacement projects. The first project involved the replacement of a brick-lined tower by an alloy acid tower for a sulphur burning plant in North America. The second project involved the replacement of a brick-lined acid tower with a NORAM designed brick-lined tower for a smelter off gas acid plant in South America. The impacts of various design considerations on acid tower replacement projects are compared, including in-situ replacement versus a new location; brick-lined versus alloy shell, and selection criteria for mist eliminators.

The sulphuric acid plant typically consists of several strong acid towers utilising sulphuric acid between 93% and 99.5% w/w concentration with temperatures ranging from 50°C to 120°C. Within this operating range, the traditional design for acid towers uses brick-lined carbon steel for the bottom section of the tower with stainless steel 316L above the acid distributor. Over the last 25 years, the use of all-metal absorption towers, where high-silicon alloys such as NORAM SX® replaced the brick-lined carbon steel section, has gained popularity as the design offers several advantages such as reduced installation time for replacement towers.

This article discusses two of NORAM's recent projects where acid towers were successfully replaced to upgrade the strong acid systems of existing acid plants, and compares some of the key tower design concepts, the selection of materials of construction and tower internals and execution strategies.

Reasons for tower replacement

Acid towers will eventually need to be replaced and upgraded due to a combination of factors including:

- plant operating conditions;
- mechanical integrity of equipment (end-of-life, corrosion etc.);
- increasingly stringent environmental regulations;
- increased production requirements.

NORAM recently completed two different acid tower replacement projects as follows:

Project A required the replacement of an acid tower for a sulphur burning plant in North America. The existing acid tower had reached the end of its service life with numerous patches on the shell, necessitating replacement. The new tower needed to accommodate operation at the current capacity for the next five to ten years after which the plant capacity is expected to increase. The new tower was also designed to:

- reduce acid mist carryover;
- reduce SO_3 slippage (based on absorption efficiency);
- reduce ammonia consumption (of a downstream scrubber system).

Project B consisted of the replacement of an acid tower for a smelter in South America. The existing acid tower had severe structural issues. The new tower was designed for:

- higher process gas flows and SO_3 concentrations;
- higher absorption efficiency;
- increased acid circulation;
- reduced H_2SO_4 and SO_3 emissions.

Fig. 1 shows the old acid towers which were replaced for the two projects.

PHOTOS: NORAM

Fig. 1: Old towers prior to replacement. Project A (left), Project B (right). Both towers had reached the end of their service life.

Design comparison

The design parameters and requirements of a replacement tower need to be agreed to and finalised at the start of any tower replacement project. There are many options for acid tower design including the selection of:

- materials of construction (brick-lined vs alloy);
- packing (type and height);
- mist eliminators (mesh pad vs candles; hanging vs standing candles; impaction candles vs Brownian diffusion candles);
- distributor (trough vs pipe);
- acid dilution (internal vs external);
- strainer (no strainer vs internal strainer vs external strainer);
- pumps (re-use vs new pumps; submerged vs external);
- piping (re-use vs new piping, alloy vs cast iron);
- ducting (re-use vs new, carbon steel vs stainless steel);
- supports and foundation (re-use vs new);
- instrumentation (re-use vs new).

The materials of construction and internals for both projects are summarised in Table 1.

Key features are described in more detail in the following sections.

Table 1: **Comparison of acid tower designs of Project A and Project**

Item	**Project A**	**Project B**
Plant configuration	single absorption	single absorption
Service	oleum absorber	absorber
Tower location	upstream of ammonia tail gas scrubber	connected to main plant stack
Size	medium (5.5 m diameter)	large (7.5 m diameter)
Material of construction	metal alloy shell	carbon steel bottom shell with brick-lining and stainless-steel top
Packing support	metallic support	self-supported ceramic dome
Packing type	2" standard saddles and NORAM 3" HP saddles	2" standard saddles and NORAM 3" HP saddles
Mist eliminator	mesh pad (co-knit)	Brownian diffusion candles (x 64)
Distributor	trough distributor	trough distributor
	522 m^3/h irrigation rate	2,000 m^3/h irrigation rate
	450 downcomers	2,200 downcomers
Acid dilution	external	internal (2 lances + crossflow)
Strainer	internal	external (3 external basket strainers)

Source: NORAM

Materials of construction

The materials of construction for acid absorption towers can be either carbon steel shell with brick-lined bottom section (below distributor) and stainless-steel top section or a combination of stainless steel and alloy for an entirely metal shell. The following requirements need to be considered:

- acid quality constraints;
- availability of specialised bricking contractors;
- available space at site for the new acid tower;
- installation time constraints and plant shut-down time;
- process design requirements (i.e. reduced emissions, increased capacity);
- project budget.

For Project A, a metal shell was selected to reduce the installation time as the on-site bricking component is eliminated. It was also designed to fit the existing foundation and to minimise modifications to the existing piping and ducting. For this alloy tower, the packing support was an alloy grid design spanning the entire tower diameter. These design decisions reduced the complexity and execution time of the project.

For Project B, a brick-lined carbon steel tower was chosen because it provides higher tolerance to acid quality variations in a smelter setting. For NORAM's brick-lined tower, the packing was supported by a self-supporting ceramic dome with an open area of up to 60% providing a very low pressure drop.

Fig. 2 shows different types of packing support utilised.

Mist eliminator

Acid towers are typically equipped with mist eliminators. The selection of the most suitable mist eliminator depends on:

- type of tower service;
- type of distributor;
- tower operating parameters;
- location of the tower;
- particle size of acid droplets.

Mesh-pads are typically used in drying tower service as the droplet size generated in drying towers is generally larger than ten microns and mesh-pads have high efficiencies for droplets larger than five microns.

Brownian diffusion (BD) candles are recommended for absorption tower service as the droplet size generated is mostly smaller than three microns in diameter.

For Project A, the old tower was equipped with a multi-layer mesh pad even though it is an absorption tower. High mist capture efficiency was not required as the tower is located upstream of an ammonia tail gas scrubber. One of the objectives of the replacement tower was to reduce ammonia consumption leading to the consideration of Brownian diffusion (BD) candles to improve mist capturing efficiency.

However, due to tower pressure drop requirements, a mesh pad (co-knit) was selected.

For Project B, Brownian diffusion (BD) candles were selected to provide the maximum practical acid droplet capture efficiency thereby eliminating acid mist emissions to the stack and to the environment.

Fig. 3 shows different types of mist eliminators.

Distributor

In both Project A and Project B, the client selected a NORAM trough acid distributor (see Fig. 4), which provides very uniform and well-dispersed acid distribution. Troughs with evenly spaced downcomer tubes distribute the acid by gravity into the packing for optimum performance and minimal spray formation. The design prevents debris in

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

PHOTOS: NORAM

Fig. 2: Acid tower packing support. Project A (left): metallic support. Project B (right): self-supported ceramic dome.

PHOTOS: NORAM

Fig. 3: Mist Eliminators. Project A (left): mesh pads. Project B (right): Brownian diffusion candles.

PHOTOS: NORAM

Fig. 4: Trough distributor. Project A (left): alloy tower. Project B (right): brick-lined tower.

PHOTOS: NORAM

Fig. 5: Acid strainer. Project A (left): internal strainer. Project B (right): external strainer.

the acid from obstructing the downcomer tubes, and the acid flow in the troughs can be inspected during operation. It is particularly suitable for large and high-performance towers.

Pyrex™ sight glasses are installed above the distributor in the tower shell and permit viewing of distributor operation.

Strainer type

Installation of a strainer in the acid circuit is recommended as entrainment of solids, such as construction debris, ceramic packing chips, breakage from brick-lined equipment, and corrosion by-products, often cause problems in acid circulation systems. Two common types of strainer are (see Fig. 5):

- acid tower internal strainer;
- external in-line basket strainer.

For Project A, an internal strainer was installed inside the acid tower at the drain nozzle. This strainer was fitted into the drain nozzle.

For Project B, an external in-line basket strainer was installed in-line with the acid piping. This strainer was fitted with a removable metal basket located at ground level allowing for easy clean out, inspection and maintenance.

Execution strategy comparison

Several strategies can be considered to execute an acid system replacement project including the requirement to keep the plant running while the equipment is installed, equipment size and weight, available space at site, installation time constraints, plant shut-down time, crane capacity, layout and ducting considerations, etc. The execution strategies for the two NORAM acid towers replacement projects are summarised in Table 2.

Installation strategies

Both towers were supplied in rings and assembled on site.

For Project A, the tower was installed in the same location on the existing foundations which minimised the modification of platforms, ducting and piping. The tower was assembled, and the packing and distributor installed inside the tower on the temporary foundations and before lifting it into place. The overall installation time of this tower was relatively fast and was completed during the plant scheduled shutdown. The installation of the tower in


PHOTOS: NORAM

Fig. 6: NORAM acid towers. Project A (left). Project B (right).

the same foundation and re-use of ancillaries allowed for significantly simpler project execution.

For Project B, the tower was assembled and installed on a new foundation, which required new platforms, ducting and piping as well as new acid pumps and external strainers. The brick lining and the installation of tower internals were completed at the tower final location. This strategy increased the overall project complexity and cost, but provided some technical advantages to meet the client requirements.

Commissioning and start-up

For Project A, the new system was commissioned during the plant scheduled shutdown. When the sulphur burning plant restarted, the acid tower and ancillaries were operated and monitored. The operation and performance of the system was confirmed as the plant was brought online.

For Project B, the new system was commissioned independently from the acid plant which was running normally using the existing tower. The new acid system including acid pumps, piping, strainers, acid circulation and distributor were operated to confirm all process parameters and to optimise the operation of the system. During a scheduled plant shutdown, the tie-ins to the new tower was completed and brought online to capture the SO_3 gases from the smelter, while the environmental performance of the new system was confirmed.

Fig. 6 shows the NORAM acid towers for both projects.

Conclusions

This article compared the design and execution strategies of two acid system upgrade projects. Some of the early design decisions have a large impact on the overall project execution, capital costs and project schedule as illustrated by two markedly different projects. Working with an experienced engineering team is invaluable to acid producers to define the most suitable design and execution strategy to achieve higher performance, and reliability at most reasonable cost. ■

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Table 2: **Comparison of acid tower execution strategies of Project A and Project B**

	Project A	**Project B**
Installation location	same location	new location
Foundation	re-use	new
Fabrication	shop fabrication of 5 rings, site assembly	shop fabrication of 9 rings, site assembly
Assembly location	temporary foundation	new final foundation
Installation sequence	assemble tower, install all internals (packing, distributor, mesh pad) and then lift to final location	assemble tower in place, brick-line and install internals in final location
Lifting	lifting tower fully loaded	lifting of rings only
Commissioning	commission after plant shutdown	commission acid circulation, distributer and strainers in parallel while plant is running (final commissioning only confirms absorption efficiency)
Platforms, ducting and piping modifications	minor	extensive
Ancillaries	re-use	3 new acid pumps, 3 new acid strainers, NORAM SX® piping system
Installation time	shorter	longer
Project complexity	lower	higher
Capital cost	lower	higher

Source: NORAM

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