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Sulphur 2018 Conference, Gothenburg Sulphur as a renewable energy carrier Sulphuric acid pumps Lean acid gas processing

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Sulphur granulation IPCO's developments for medium

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An uncertain world

s the sulphur industry meets in Gothenburg this November for the 2018 Sulphur Conference, the future for the industry, and perhaps even the world in general, looks more uncertain than it has for some time. Various factors are driving this – technological, environmental, economic but above all political, because like it or not, it is politics that drives a lot of the major commodity markets as much as economic or other factors.

The interplay of these factors can be seen in some of the major imponderables for sulphur markets. The impact of the forthcoming IMO 0.5% sulphur cap in marine fuels, for example, is ostensibly an environmental issue, but technological and economic factors play into whether ship owners buy low sulphur fuels or install scrubbing systems. As the overwhelming evidence seems to be that they will look to low sulphur fuels, especially distillates, so refiners have to start looking to see if they can capture value from this, while meanwhile there are forecasts that the price premium for low sulphur crude - already wide - may widen stull further, and make, according to one report, up to 25% of Canada's oil sands uneconomic, at least until the market rebalances a few years down the line.

The uncertainty is also reflected in the fact that sulphur prices themselves have become more volatile. The end of 2017 and the start of 2018 saw prices push towards and even past \$200/t, before falling back to \$120/t, and now rising again back towards \$150/t f.o.b. Arab Gulf. Some of this is also down to technological or economic factors - the rise of huge sour gas projects in China, the Middle East and Central Asia have meant that single projects can now represent over 1 million t/a of additional sulphur supply, and delays or operating difficulties thus have a concomitantly larger impact on market supply than smaller projects would do. But once again politics play a part - renewed US sanctions on Iran, the ongoing China-US trade spat, attempts to reformulate NAFTA or the UK's impending departure from the EU all add uncertainty. Likewise China's push towards more environmental responsibility from its heavy industries - enforced by heavy-handed blanket bans on production, mandated moves of entire production sites, and caps on use all combine with attempts to rationalise overcapacity in key industries like coal and steel, and speak to a major restructuring of the economy

which has driven commodity markets for the past twenty years. China has been the largest importer of sulphur for some years, but as its phosphate sector rationalises, new sour gas capacity boosts domestic sulphur production and acid from new copper smelters displaces the need for so much sulphur-burning acid capacity, China's imports seem very likely to fall.

What can be done to mitigate, or at least hedge against such uncertainty? The answer that both the world's largest producer and the world's largest consumer of sulphur – Adnoc and OCP respectively - have come up with is to deepen ties between producer and consumer and set up a long term supply arrangement on a more predictable price basis. Given that Adnoc supplied 2 million tonnes of sulphur to Morocco in 2017, the attractions of a more predictable trading basis must be considerable in today's market. Indeed, last November the companies moved further, by setting up a joint venture company to develop new fertilizer capacity. It is a significant move downstream for Adnoc, and could presumably involve the company in owning phosphate capacity which it is the primary sulphur supplier for. Given OCP's current focus on developing the African fertilizer market, which has the largest potential for growth of any region over the coming decades, this could be a way into that untapped potential for Adnoc. It also has echoes of Mosaic's tie-in with Ma'aden in Saudi Arabia.

It is impossible to control markets, governments or technical developments, but it is possible to develop stronger direct business relationships between producers and consumers to help take some of the sting out of market swings, and it is a good bet that more of these global industry partnerships will develop over the next few years.



Richard Hands, Editor

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Sulphur prices themselves have become more volatile.

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MARKET INSIGHT

Meena Chauhan, Research Manager, Integer Research (in partnership with ICIS) assesses price trends and the market outlook for sulphur.

SULPHUR

Sulphur prices have remained well above \$100/t f.o.b. across major exporting regions through 2018 so far, and firmed further going into September. Regional tightness has been a key market bull factor this year as outages, sweeter crudes at refineries and logistical constraints have reduced availability. Demand has been healthy in North Africa and Latin America, with import trade ticking up to reflect this. Tightness in the sulphuric acid market due to major smelters being offline has also lent some support to sulphur market.

Middle East producer sulphur prices for August and September 2018 were posted at increases. In Qatar, Muntajat announced its September Qatar Sulphur Price (QSP) at \$145/t f.o.b., representing an 8% rise on a month earlier, up by US\$11/t. This price level had not been seen since December 2017. In Saudi Arabia, the August price was announced at \$138/t f.o.b. by Saudi Aramco Trading. At the time of publication, the September price was expected to be above \$150/t f.o.b. but was not confirmed. Adnoc in the UAE posted \$140/t f.o.b. Ruwais for tonnes being lifted for the Indian market in August. Middle East supply has been on the balanced to tight spectrum for much of the year - leading to stable to firm pricing.

Logistical challenges for shipments out of the Black Sea have not eased, with vessel availability heard to be an issue through to the third quarter. As we approach the winter period, a further slowdown will emerge once the seasonal closure of the Volga Don waterway is underway. A factor expected to support firm pricing through the remainder of the year.

Sulphur inventories at the nine major ports in China have been a talking point for most of the year, as stocks grew to 1.7 million tonnes in August before dropping dramatically down to 1.2 million tonnes by the end of the month. Despite some buyers retreating to the sidelines, prices in China ticked up through the third quarter of the year and into September. The appreciation of the Chinese Yuan was attributed to some of the increases in July/August, rather than a meaningful rise in demand. Prices were up at \$164/t c.fr in August, compared to the lowest average in the year at \$134/t c.fr back in February. At the start of September, offers of around the \$170/t c.fr level were heard but not confirmed. As inventory levels at the ports began to erode in August this added to the bullish sentiment for the short term pricing outlook in the market. Trade data for sulphur imports to China remains limited to the first quarter of the year - which showed imports dropped 14% to 2.6 million tonnes - a low not seen since the same period in 2015. Beyond the first guarter, market sources have speculated that imports are likely to remain below year ago levels. Local sulphur production has been healthy through the year, and the rise of refining projects and developments at sour gas fields are bears for the future of trade into China.

Several supply additions expected for 2018 have been delayed for the most part, further adding support to firm pricing. However, over in Kazakhstan, the Kashagan project is estimated to be at 80-90% of capacity in 2018, and expected to reach its capacity of around 1.2 million tonnes per year in 2019. Other projects in the pipeline set to impact supply include the long delayed Rasgas Barzan project in Qatar – with total potential sulphur output of around 850,000 t/a at capacity. A time-line for the project remains unclear with a tender for a gas pipeline closing earlier this year potentially adding to the delay.

Brazil remains a key market for sulphur out of the US Gulf, with around 500,000 tonnes shipped in Jan-July 2018, on a par with year ago levels. There has been a dramatic shift in trade from Russia to Brazil meanwhile, with imports more than doubling to over 400,000 tonnes, representing ~28% of total trade into the country. Brazilian prices firmed to \$/160/t c.fr in August on the high end, from lows of \$129/t earlier in the year. A fire at Petrobras' 415,000 bbl/day Replan refinery in August temporarily reduced production by 50% but a restart was planned for the start of September and not expected to significantly impact sulphur output.

Fourth quarter domestic contract negotiations in NW Europe are set to begin in September, but early indications are for a rollover or slight increases. The supply/demand balance in Europe has been tight owing to high demand from the downstream markets with improved economics across the chemicals and industrial sectors. Despite the usual seasonal slowdown through the summer, market sources reported strong demand. In Germany, supply has been tight due to issues at Gros-



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senkneten. A restart had been expected in August but remained in question at the start of September. This alongside refineries heard favouring sweeter crudes is likely to lead to a persistent tight market in the coming months and potentially into the new year.

The global market in 2018 is expected to remain largely in balance owing to project delays, supporting prices across major benchmarks for the coming months. A market surplus had previously been expected this year but this is now forecast to emerge in 2019. Healthy demand across downstream markets and the ongoing tightness in sulphuric acid is also aiding the outlook for the upside in the market for the short term. The medium term view remains for length due to the rise of new projects across the energy sector.

SULPHURIC ACID

Global sulphuric acid prices continued on an upward trajectory through the second and third quarters of the year with no signs of abating at the start of September. The tight market balance is expected to fuel prices through to Q4 and potentially into the new year.

NW European spot prices for the export market jumped from lows of \$33/t f.o.b. to highs of \$75/t f.o.b. between Q1 and Q3 2018. The price rise has been buoyed by tight supply at smelters, with major producers reporting low inventories and strong demand. Downstream markets across key industrial and chemical sectors in Europe have been running at healthy capacity rates this year, leading to improved offtake of raw materials, including sulphuric acid. Smelter outages in other regions have led to increased spot demand for European acid volumes and is expected to remain a factor in the pricing outlook in the short term.

Smelter acid issues in Asia have been a driving factor for trade and pricing this year. In India, the Vedanta/Sterlite Tuticorin smelter closure has led to disruption in the market. At 1.2 million t/a of sulphuric acid capacity, the stoppage left offtakers to source volumes from alternative suppliers. While buyers in India outside of the Tamil Nadu region have not been directly impacted by the outage, tightness has led to firmer pricing and reduced availability in the spot market. Some market sources expect the smelter will eventually come back online, likely in 2019. Producer Vedanta has challenged the closure order and subsequently the National Green Tribunal (NGT) formed an independent judicial committee set to decide the fate of the smelter. The NGT's report was due to be submitted in September. A potential restart would ease pressure in India and likely prices would follow.

Moroccan sulphur and sulphuric acid imports remain a major market focus due to the impact this could potentially have on the smelter acid balance and prices. OCP has continued to ramp up its processed phosphates expansion at Jorf Lasfar, with increased sulphur based acid production capacity. In 2017, sulphuric acid imports reached record highs at 1.5 million tonnes for the year. In 2018, trade has continued to rise, reflecting increases beyond levels a year earlier. In official data for Jan-Jun 2018, acid imports totalled 875,000 tonnes – a 23% increase year on year. Market sources also indicate year to date shipments have exceeded 1 million tonnes, with some estimating acid imports to increase further – speculating this could be closer to the 2 million tonne mark for the year.

Demand for imported acid in Brazil has been lacklustre so far in 2018, owing to high prices – up at around \$110/t c.fr in August. End users have been reluctant to accept inflated prices, as downstream product margins were squeezed.

Elsewhere in Latin America the situation has been markedly different. In Chile, acid demand has been spurred by higher operating rates at copper mines on the back of improved copper prices at the start of the year. At the same time, industrial action led to some local supply disruption, leading buyers to enter the spot market. Trade data shows how this has contributed to the price run up in the international market. Acid imports to Chile in the first half of the year totalled 1.4 million tonnes - up 23% compared with 2017 and also significantly above 2016 levels. While Peru remains the leading supplier to Chile, there has been a leap in imports from Japan and South Korea - totalling over 400,000 tonnes so far.

Price indications

Cash equivalent	March	April	Мау	June	Jul
Sulphur, bulk (\$/t)					
Adnoc monthly contract	140	127	125	136	13
China c.fr spot	135	135	152	157	16
Liquid sulphur (\$/t)					
Tampa f.o.b. contract	116	113	113	113	12
NW Europe c.fr	112	117	117	117	n.a
Sulphuric acid (\$/t)					
US Gulf spot	95	95	100	100	11(

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Market outlook



SULPHUR

- Brazil and Morocco have been stable spots for demand so far this year and are likely to remain key outlets for the remainder of the year, supporting trade and pricing.
- Developments in China will remain a focus in the outlook. Increased domestic production from refineries and gas projects is raising question marks over the future of imports to the country.
- The disruption to Syncrude's operations in Western Canada is expected to improve during the third quarter, with sulphur production to return to normal rates.
- Upcoming IMO 2020 regulations may influence upgrade projects across the refining sector, although there is no industry consensus on how the shipping sector will meet the changes. Some refiners have already implemented upgrades, resulting in an uptick in sulphur recovery while others continue to assess the market in the interim.
- **Outlook:** Sulphur prices are likely to remain firm in the short term due to the

regional supply issues as well as the approach to the winter season. In Canada and Russia, logistics can be impacted by weather during this period. At the same time, buyers may start to resist continued price increases ahead of fourth quarter contract negotiations unless these can be passed onto respective downstream markets. Longer term, increased sulphur recovery from major projects in the Middle East, FSU and Asia are likely to dampen the upside to pricing.

SULPHURIC ACID

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- China smelter acid production is ramping up, and there has already been a significant upturn in offshore exports in 2018. Historically there has been one major Chinese acid exporter. However, at least two metallurgical acid producers are now offering tonnes in the export market – likely paving the way for a trend that may soften prospects for pricing in the long term.
- China acid imports are expected to show a decline for 2018, although limited trade data is available. The rise in domestic output and slowdown in

demand is reported to be taking a toll on shipments from North East Asia.

- There are limited planned turnarounds at smelters in the first half of 2019 – pointing to the potential for a downward correction pricing, should supply start to normalise in Asia.
- Glencore's PASAR Philippines smelter is set to see improved operating rates in 2019 following the turnaround in Q4 that will see the cooling towers at its sulphuric acid plants replaced, adding to the potential for a more bearish market.
- **Outlook:** There appears to be limited downside for the acid market in the short term outlook. Supply is tight in Asia and Europe. A question mark hangs over the future of the Sterlite Tuticorin smelter in India. Demand has improved in Chile, with buyers in the copper sector absorbing increased volumes of acid. Morocco's appetite for acid only appears to be growing. These factors point to firm prices for the rest of the year. Higher elemental sulphur prices may also lend support for contract prices to see an uptick in the fourth quarter.

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Sulphur Industry News

CHINA

China imposes tariffs on US oil products

China has implemented tariffs on a second tranche of US goods, targeting oil products and coal for the first time, in retaliation to US tariffs. Energy commodities including propane, butane, naphtha, jet fuel and coal are on the second list of \$16 billion worth of US products that attracted 25% additional tariffs from August 23rd, according to China's Ministry of Commerce. The first tranche of tariffs implemented on July 6th saw China retaliate by imposing a 25% tariff on \$34 billion worth of US imports of food products and agricultural commodities such as soybean and automobiles. The upcoming third round of US tariffs is on \$200 billion worth of goods, at a lower rate of 10%. The Office of the United States Trade Representative set August 30th as the final date the consultation process. Chinese retaliation could cover crude oil and LNG exports from the US. US exports of petroleum products averaged 230,000 bbl/d in 2017 and 180,000 bbl/d in 2016, but were down to 140,000 bbl/d in May 2018.

KUWAIT

Al Zour refinery contracts awarded

The Kuwait Integrated Petroleum Industries Company (KIPIC), a subsidiary of the stateowned Kuwait Petroleum Corporation, has awarded SNC-Lavalin a \$180 million contract for management support services, as well as the preparation and delivery of training, documentation and competency development consultancy services at the Al Zour refinery in Kuwait. Under the agreement, SNC-Lavalin will provide commissioning technical services including master plan, start-up program development, risk assessment and management, commissioning management support, operations readiness and assurance, project phase execution activities, as well as training, competency development and assurance, documentation preparation, and the development of a knowledge management system and e-learning services, software, procedures and conducting a safe and efficient start-up and operations.

"We look forward to building a long term relationship with KIPIC and working in association with them to commission one of the newest and biggest refineries in the world. This is a great opportunity to further demonstrate our globally renowned expertise and extensive capabilities in downstream, and to support our client in executing their strategy and delivering a successful start-up and the steady state operations of the refinery," said Christian Brown, President, Oil & Gas, SNC-Lavalin.

The 615,000 bbl/d Al-Zour refinery will be the largest in the Middle East, increasing Kuwait's refining capacity to more than 1.5 million bbl/d. The refinery will predominantly produce low sulphur fuel oil to replace high sulphur fuel oil currently used in local power generation plants. Other refinery end products will include ultra-low sulphur diesel and kerosene, petrochemical naphtha, granulated sulphur and LPG. It is due for start-up in 2022.

DuPont Clean Technologies says that it will also provide its *STRATCO*[®] alkylation and *MECS*[®] sulphuric acid regeneration (SAR) technologies for the refinery. The alkylation unit will have a capacity of 9,100 bbl/d and the acid regeneration unit 70 t/d. The alkylation unit will feature the *Contactor*[™] XP2 technology. This patented reactor enhancement makes efficient use of the tube bundle heat transfer area, ensuring the highest quality alkylate product.

"The new alkylation and MECS advanced SAR units will help KIPIC to fulfil its ambitious target for desulphurisation," said Eli Ben-Shoshan, global business director, DuPont Clean Technologies. "This is the first MECS advanced SAR license... we are delighted to be supporting KIPIC in making a project that is of such vital importance to Kuwait's national refining capacity sustainable with our clean air and clean fuel technologies."

Gas sweetening project to be re-tendered

The Kuwait Oil Company (KOC) has decided to retender a contract to build a gas sweetening facility in western Kuwait. The contract was originally expected to be signed in September, with Kuwait-based Spetco International Petroleum tendering the lowest bid at \$277 million. KOC not divulged why the tendering process was cancelled, but has asked contractors to express interest in the new tender. Only two of the 29 pre-qualified companies actually submitted commercial bids to the original tender.

The original contract consisted of the 120 million scf/d gas facility itself (running under an engineering, procurement and construction contract) as well as a twin train 200 t/d sulphur recovery unit which would be constructed on a build, own, operate (BOO) model. The gas plant would take sour gas from upstream processing units, at about 4% H_2S and 10% CO_2 concentration and would use amine solutions for sweetening, with zero flaring.

CANADA

Oil sands production projected to grow

Canada's oil sands will experience large production growth through 2019 followed by more modest, steady growth through 2027, according to a new 10-year production forecast by IHS Markit. The forecast is for production to rise more than 500,000 bbl/d in 2019 and up to 1.0 million bbl/d by 2027 compared to today. Canadian oil sands have gained importance to the heavy oil market as the only source of material supply growth in the world for that type of crude. Output from other large producers of heavy oil, most notably Venezuela, where production has fallen by more than one million barrels per day in recent years and is expected to fall further, has declined.

The strong growth in the near term is expected to come from the completion of projects sanctioned prior to the oil price collapse, the revival of some deferred projects as well as some new investments in capital efficiency projects. Following 2019, uncertainties related to much-needed infrastructure, particularly pipeline capacity, point to a deceleration of growth, IHS Markit says.

Kevin Birn, IHS Markit North American Crude Oil Market Team director said: "Pipeline constraints have exacerbated price discounts for Western Canadian heavy oil relative to global benchmarks. Over the past 12 months alone, the difference in price compared to a barrel of West Texas Intermediate (WTI) has fluctuated just under \$10/bbl to more than \$30/bbl. This sort of price volatility is weighing on investment decisions in western Canada and will likely continue to do so until greater certainty can be achieved."

Nevertheless, the outlook does continue to project growth in part due to the unique nature of oil sands projects, which do not experience production declines meaning that any incremental invest-

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ment can add to existing production and contribute to growth. This represents a strategic advantage for oil sands asset owners coming out of a low-price period in that there is no production deficit to overcome. IHS also expects greater crude-byrail movements to help pick up the slack in the interim and for new pipelines to be built eventually.

Jacobs wins sour gas processing contract

Jacobs Engineering Group Inc. has secured a contract to provide engineering services for Keyera's Wapiti Gas Plant Phase Two expansion, adding 150 million ft³/d of sour gas processing to the plant currently under construction near Grande Prairie, Alberta, Canada. Keyera estimates construction of this second phase will be completed by mid-2020 at a total installed cost of US\$150 million. Jacobs also engineered the first phase of the gas plant in the liquids-rich Montney region of northwestern Alberta. Once both phases are in operation the facility will have a capacity of 300 million scf/d of sour gas and 25,000 bbl/d of condensate.

CASPIAN SEA

International settlement on Caspian Sea status

The five states which border the Caspian Sea; Russia, Iran, Azerbaijan, Kazakhstan and Turkmenistan, have signed a landmark international convention on the legal status of the body of water. The agreement establishes a formula for dividing up its resources and prevents other powers from setting up a military presence there.

At its heart was whether the landlocked, freshwater body is defined as a sea or a lake. The USSR and Iran had previously agreed on its status as a lake, with the USSR claiming the northern two thirds or so, but the breakup of the Soviet Union in 1991 and the creation of several new states led to new claims, while Iran has tried to maintain the original status as a lake. The difference is that seas are covered by international maritime law, specifically the United Nations Law of the Sea. This sets established rules on management of natural resources. territorial rights, and the environment. It also allows states which do not border the sea to have free access to resources there. On the other hand, if it is defined as a lake, then it must be divided equally between all states that border it. This would have given Iran greater access to undersea bodies of oil and gas. It is estimated there are 50 billion barrels of oil and nearly 300 trillion cubic feet (8.4 trillion cubic metres) of natural gas beneath its seabed.

As a compromise, the convention signed in the Kazakh city of Aktau in August gives the body of water a "special legal status" which means it is neither a sea nor a lake. The surface water will be in common usage, meaning freedom of access for all littoral states beyond territorial waters. However, the seabed mineral rights will be divided up. But, because it is not a lake, this division does not need to be equal, and seabed boundaries remain to be negotiated – via bilateral agreements.

The disagreement over its legal status has prevented a natural gas pipeline being built across the Caspian between Turkmenistan and Azerbaijan. This would have allowed Turkmen gas to bypass Russia on its way to Europe.

IRAN

Iran bans import of oil industry equipment

The Iranian Oil Ministry has passed an order to ban the import of 84 items, including any oil industry equipment which can be produced inside the country, according to Iranian media sources. The move, designed to support Iranian-made products and maximise the use of domestic products and services, prohibit companies and subcontractors from purchasing wellhead equipment, desalination packages, anticorrosion agents, sulphur recovery catalysts, wellhead control panels and many other items from overseas.

SRU to start at Ilam by end 2018

The managing director of Persian Gulf Petrochemical Industries Co. (PGPIC), Jafar Rabie, has told Iranian media that the sulphur recovery unit at the Ilam Petrochemical Company – due to be on-stream in March this year – will now be operational by the end of the year. Ilam is building an olefin plant to process 450,000 t/a of ethylene and a 400 t/d sulphur recovery unit.

Meanwhile, Iran's President Hassan Rouhani has officially inaugurated Phase 2 of the Gulf Star Refinery at Bandar Abbas. With the launch of the project, the refinery's production capacity of Euro-V standard gasoline will double to 150,000 bbl/d. The third and final phase of the expansion project is expected to come on stream by the end of the current Iranian year (March 2019), taking Euro-V gasoline capacity to 225,000 bbl/d, as well as a total of 130,000 bbl/d of light and heavy diesel, jet fuel and gas condensates. Sulphur production will increase to 130 t/d, and a new granulation plant forms part of the project.

UNITED STATES

Delaware City looks to process more sulphur

PBF Energy, the owner of the Delaware City refinery, says that it is constructing a new \$100 million hydrogen plant at the refinery, to be operational in late 2019. The company aims to capitalise on a need for low sulphur diesel in the wake of the International Maritime Organisation's 0.5% cap on sulphur content of bunker fuels from 2020. The hydrogen plant will allow Delaware City to expand the output of its coking section and generate an additional 7-10,000 bbl/d of low sulphur diesel, but primarily it will allow the facility to process more heavy, sulphur-rich crudes. The company has an existing permit to remove up to 20,000 t/a of sulphur from refinery products.

INDIA

Black and Veatch to build SRUs for refinery project

Black & Veatch has been selected to provide process technology and the basic engineering design package for the sulphur recovery portion of the grassroots Eastern India Refinery Project planned by the Indian Oil Company (IOC). The new sulphur recovery facilities will comprise three Claus sulphur recovery units (SRUs), each with a capacity of 525 t/d, as well as two 1,050 t/d tail-gas treating units. The sulphur recovery complex is being designed using Black & Veatch's two-bed Claus and tailgas treating technologies. Overall sulphur recovery is guaranteed at 99.9% minimum under the full range of expected operating conditions. The new SRUs will process amine acid gas and ammonia-bearing sour water stripper acid gas from conventional refinery systems. Degassing technology is also included to reduce total H₂S (hydrogen sulphide) content in the molten sulphur to less than 10 ppmw.

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JDCPhosphate demonstrates continuous phosphoric acid process

Florida-based JDCPhosphate, Inc says that it has successfully used its proprietary Improved Hard Process technology to produce high quality super-phosphoric acid (SPA) using low quality phosphate rock tailings as feedstock without creating toxic phosphogypsum waste during continuous operation of its new demonstration plant in Fort Meade, Florida. The Improved Hard Process (IHP) is a kiln-based process that avoids direct acidulation of phosphate rock, minimising the amount of waste and completely avoiding phosphogypsum production. Instead, it coproduces a commercially useful aggregate for construction and road building called *J-Rox*.

During recent operations at its demonstration plant, JDC was able to operate its entire process continuously - including feed preparation and agglomeration, induration, reduction, oxidation, and acid production - to produce super-phosphoric acid using phosphate rock waste tailings from local mining operations. The phosphate raw material contained an average of about 14% phosphate as P205, with high levels of silica and other impurities, including magnesium oxide. The company combined this low quality phosphate material with clay and petroleum coke to make its kiln feed. The feed was then processed in the kilns and acid plant to produce super-phosphoric acid with a concentration of 68% P205 with less than 2.5% impurities, including less than 0.3% calcium, 0.2% magnesium, 0.3% sulphate, 1.0% iron oxide, 0.1% F and 0.1% aluminium. The SPA also contained about 2 ppm cadmium, with about 80-90% of cadmium in the phosphate feed being eliminated during the process and captured in pollution control scrubbing systems. The process also significantly reduces levels of other trace heavy metals, such as lead and arsenic.

"This is a major milestone for JDC and our technology, showcasing IHP's value as a cost-efficient and scalable new process," said Timothy Cotton, CEO of JDCPhosphate. "Our company, management team and stakeholders have dedicated a great deal of time and effort to prove the efficacy of this breakthrough process and we are very pleased with these recent achievements. Given the limited phosphate rock reserves in the world, it will be critical for future generations that we waste as little as possible of these vital resources. At the same time, we need to minimise the production of toxic phosphogypsum wastes and reduce the level of harmful impurities in phosphate products. The IHP technology will become a critical part of the global phosphate production chain."

Luc Maene, former director general of the International Fertilizer Association (IFA), commented, "For many years I have been hoping that innovative technologies will improve the sustainability of the phosphate sector, which is so critical for food production and so dependent on a limited natural resource. JDC's IHP technology can make a major impact on phosphate sustainability by opening up new sources of phosphate rock while significantly reducing wastes. This squarely addresses some of the most pressing issues confronting the industry."

Over the next few months, JDC will further upgrade its commercial demonstration plant for on-demand and sustained operations. By early 2019 the Fort Meade plant will be capable of testing various qualities of phosphate ore raw material, allowing potential licensees to validate the process for the phosphate ore and silica sources they have available. JDC will then complete its process design engineering for commercial-scale applications of the IHP technology. The company also is exploring optimal routes to commercialisation of IHP, including expanding its operations in Florida.

CANADA

Arianne signs MoU for supply of sulphuric acid

Arianne Phosphate, a Canadian-based company which is aiming to develop the Lac à Paul phosphate deposits 200 km north of the Saguenay/Lac St. Jean area of Quebec, says that it has signed a memorandum of understanding for the supply of sulphuric acid, as part of an ongoing in-house review of the project, pending a final decision to proceed. The project would include a phosphoric acid plant capable of producing high purity phosphoric acid for both domestic and international markets. The company says that several potential sites have been reviewed and further work is currently being undertaken to try and finalise a potential location, getting a better understanding rof the logistics, economics and end-product specifics. Arianne says that it will "require a

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supply of sulphuric acid that could be consistently delivered in timely fashion", and on this basis it has entered into an MoU "with a large marketer of sulphuric acid. This agreement will allow the company to work with a strong industry player that can provide the venture ongoing guidance on pricing, logistics and industry supply/ demand trends; all necessary aspects in determining the project's viability."

Wildfires temporarily idle lead smelter in British Columbia

Teck Resources has was forced to idle lead smelting operations for seven days at its Trail Operations in British Columbia, because of the effect of dozens of wildfires burning across the Canadian province following a long run of hot, dry weather. According to the company, the flash smelter requires high purity oxygen, which could not be supplied due to issues with the air quality in the on-site oxygen plant. Zinc smelting operations were unaffected. Teck produces approximately 90,000 t/a of refined lead and 295,000 t/a of zinc at Trail. Teck is halfway through building a second acid plant at the site to process sulphur dioxide off-gases from smelting, which will replace two older units at the site.

AUSTRALIA

Olympic dam smelter down due to issues with acid plant

During the release of its annual results, BHP has said that ore processing at its Olympic Dam copper, gold and uranium mine in South Australia is being affected by an ongoing technical issue, caused by the failure of several boiler tubes at the acid plant. The company did not give any timescale for the resolution of the problem, only stating that "remediation and mitigation activities are underway, and underground mining operations continue as normal." It is believed that up to two months of production could be lost at the site.

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SULPHURIC ACID NEWS



The Sterlite copper smelter, India.

INDIA

Sterlite copper closure still contested

The Indian government has backed a local decision to permanently close the Sterlite copper smelter at Thoothukudi. Initially closed by order of the Tamil Nadu Pollution Control Board, which declined to renew the plant's license to operate, this was followed by a national government order issued by the Environment and Forests department for permanent closure of the plant and remediation of pollution there. Around 1,300 tonnes of sulphuric acid has been drained from tanks at the site and removed in 75 tankers following a suspected leak. The plant has long been a bone of contention with the local community due to alleged sulphur dioxide pollution, culminating in violent public protests in May which led to 13 demonstrators being shot.

However, operating company Vedanta is still contesting the decision to close the site, with hearings continuing in August. In early August the National Green Tribunal (NGT) - India's environmental court allowed the company access to the site for "administrative purposes", and the NGT said on August 20th that an independent judicial committee would decide whether to allow Vedanta to reopen its copper smelter, with a decision expected to take up to six weeks. The committee includes representatives from Vedanta, the Tamil Nadu Pollution Control Board and the federal Environment Ministry, and will look into the matter afresh with an outcome that potentially could overrule all previous decisions. Vedanta says that the closure of the site will cost the company \$100 million per year in lost income.

ZAMBIA

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Nkana to recover copper from acid leach of slag

The Nkana Alloy Smelting Company has set aside over \$40 million to recover copper and cobalt from waste at their Kalulushi

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site, according to the Zambian Daily Mail. The project will involve recovering of copper and cobalt from slag, which will be sourced from the slag dump situated south of Kitwe. According to the environmental impact assessment report submitted by the company to the Zambian Environmental Management Agency, the slag has an average copper content of 1.23% and cobalt content of 0.89%. The slag will be crushed and ground, soaked, and then acid leached before purification and electrowinning.

CHINA

Copper prices tumble due to US-China trade war fears

Copper prices have dropped nearly 20% from their peak in June of \$7,200/t to below \$5,900/t in August because of fears of lower demand caused by the current US-China trade spat. China represents about half of all global copper demand, and although refined copper demand is estimated to have risen about 1% to April compared with the same period for 2017, according to the International Copper Study Group, this masked a 3% fall in Chinese demand as the economy slows. News of a deal between workers and operators at the Escondida copper mine in Chile, where there had been threats of a strike at the world's largest copper mining operation, have also served to lower expectations of supply disruptions and helped prices fall further.

UGANDA

Sukulu phosphate plant to open in October

The Sukulu phosphate production plant will become operational in October, its Chinese developer has said. The completion date was confirmed by Jane Guo, CEO of Guangzhou Dongsong Energy Group, the company behind the project. According to local media reports, Ms Guo said fertilizer production will officially begin on 9 October – Uganda's Independence Day – and then ramp-up to full production by June next year.

The 300,000 tonne capacity Sukulu phosphate plant is being built on a 600acre site in Uganda's Tororo district. Developer Guangzhou Dongsong also holds a license to mine the phosphate rock required for the plant from the Osukuru Hills. The project is backed by \$650 million of finance secured through the Industrial and Commercial Bank of China. "We are on course. Different sections have been given to different contractors and so far the progress is good. The project is good," Guo said, speaking to journalists on 20 July.

Agronomic trials and model farms in Kabale, Mbale and Masaka had delivered impressive results, according to Guo. She also stressed that the plant's phosphate fertilizer output would be entirely organic. "Our fertilizer is designed for Ugandan soil, climate and environment. We are purely organic," Guo said.

The project has experienced a number of delays since it was officially launched by Uganda's President Museveni in August 2014. The fertilizer plant was due to be completed in March under the latest timetable. Guo blamed rigorous testing of the production technology and financing hold-ups for project delays. She now expects the completion of the entire industrial park in June 2019.

"We had technological and financial challenges. It has taken us three years to develop the modern technology that we are going to employ in the project. We also got the funds last year. Construction started in February this year and we have the infrastructure for the entire plant," Guo said.

PERU

Mina Justa secures financing

Peruvian mining company Minsur says that it has secured over \$800 million of financing from a consortium of international banks, and aims to begin construction work this year on the \$1.6 billion Mina Justa copper mine. The mine, in Peru's Ica region, will produce 100,000 t/a of copper at capacity. The company raised a further \$450 million in 2014 from a bond issue. South Korea's state-run export credit provider Export-Import Bank of Korea (Korea Eximbank) agreed to finance \$200 million of the required loans to help ensure a steady supply of the key mineral for Korean companies. Minsur is the mining arm of Peru's largest conglomerate Breca, and Chile's largest refining company Copec. Also involved in the project is LS-Nikko Copper, a South Korean-Japanese consortium, which signed a deal to purchase 30% of the copper output over the next 10 years once it begins production in 2021. LS-Nikko Copper, an affiliate of Korean conglomerate LS Group, is the only copper smelting company in Korea. Other finance came from banks based in Korea, Germany, Australia and Canada.

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People

Dave Hans has rejoined Tiger-Sul Products as marketing manager. Hans worked at Tiger-Sul from 2012 to 2016 leading the company's marketing and branding program. According to a company news release, his role will help the company expand to new international markets and better serve its existing markets.

"Dave Hans is a tremendously experienced marketer, and we're excited to welcome him back to Tiger-Sul," company president and CEO Don Cherry said in a news release. "Dave's return is an example of our ongoing commitment to educating the industry about the important role sulphur fertilizer plays in modern crop production, and how growers can use Tiger-Sul products more effectively to improve yields and return on their fertilizer investment.

SQM'S CEO **Patricio de Solminihac Tampier** has resigned and will be replaced by Ricardo Ramos Rodríguez from the start of 2019. The company's board expressed its appreciation of Mr de Solminihac's contribution to the company stretching back more than 30 years.

"When Patricio de Solminihac began his career as an executive at SQM in 1988, the company had assets totalling approximately \$152 million, today SQM has assets of over \$4.3 billion," said Alberto Salas, chairman of SQM's board, adding: "It is difficult to summarize in a few words the achievements of Patricio. We regret his resignation, and appreciate his dedication and commitment over the past years."

In response, Mr de Solminihac said: "I would like to thank the entire team of over 5,000 employees in Chile and abroad for supporting me over the past three decades."

Incoming CEO designate Ricardo Ramos Rodríguez has been with SQM for over 29 years. He is currently chief financial officer (CFO) and vice president of corporate services.

"During this period, Ricardo Ramos has worked alongside Mr de Solminhac for over 20 years, which will ensure the continuity of the direction of the company, and ratifies the intention of the board to maintain the strategic path given by the current CEO," Alberto Salas said.

Sergey Momtsemlidze became Uralchem's new CEO in August, replacing Dmitry Konyaev who was named deputy chairman and will take control of strategic development. In a statement, Uralchem praised Dmitry's contribution to the company: "Since 2011, when Dmitry Konyaev took over the CEO position, the company has significantly strengthened its position on the market. The commercial output increased from five million tonnes in 2011 to 6.3 million tonnes in 2017. The sales market now includes 80 countries (compared to 50 before), while the company's product portfolio has expanded by 2.3 times – from 30 to over 70 product grades, primarily due to highly efficient and innovative types of fertilizers."

Incoming CEO Sergey Momtsemlidze was formerly the director of Uralchem's KCCW Branch in Kirovo-Chepetsk, the largest enterprise within the company. He will focus on the improving the operational activities and the efficiency of the company's business. During a seven-year tenure at KCCW Branch, Sergey invested RUB14.3 billion in production, increasing commercial output by 13 percent and average labour productivity by over 12 percent, while at the same time reducing unscheduled downtimes by more than five times. The construction of a large production plant for Uralchem's new high-purity and much-in-demand calcium nitrate product was a particular success.

Mr Momtsemlidze a graduated in Industrial Economics and Management from Perm State Technical University and holds an MBA from Russia's National Economy Academy.

Calendar 2018/19

OCTOBER

T.B.A.

Brimstone STS Advanced Sulphur Recovery Seminar, HOUSTON, Texas, USA Contact: Mike Anderson, Brimstone STS Tel: +1 909 597 3249 Email: mike.anderson@brimstone-sts.com Web: www.brimstone-sts.com

14-17

Middle East Sour Plant Operations Network (MESPON), ABU DHABI, UAE Contact: UniverSUL Consulting, PO Box 109760, Abu Dhabi, UAE. Tel: +971 2 645 0141 Fax: +971 2 645 0142 Email: info@universulphur.com

NOVEMBER

5-8 Sulphur 2018 Conference, GOTHENBURG, Sweden Contact: CRU Events Tel: +44 20 7903 2167 Email: conferences@crugroup.com

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European Refining Technology Conference, CANNES, France Contact: Sofia Barros, Senior Conference Producer & Project Manager, World Refining Association Tel: +44 20 7384 7944 Email: sofia.barros@wraconferences.com

FEBRUARY 2019

4-5

SulGas Gas Treating & Sulphur Recovery Conference, MUMBAI, India Contact: Conference Communications Office, c/o Three Ten Initiative Technologies LLP, 12-1-16 Waltair Main Road, Visakhapatnam, Andhra Pradesh, India. Web: www.sulgasconference.com

25-28

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

MARCH

17-19

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

25-27

Phosphates 2019 Conference, ORLANDO, Florida, USA Contact: CRU Events, Chancery House, 53-64 Chancery Lane, London WC2A 1QS, UK. Tel: +44 20 7903 2167 Email: conferences@crugroup.com

APRIL

16-17

Sulphur World Symposium, PRAGUE, Czech Republic Contact: The Sulphur Institute Tel: +1 202 331 9660 Email: sulphur@sulphurinstitute.org

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Using sulphur to store renewable energy

Overcoming the problems with storage of intermittently generated renewable electricity requires converting it to a form in which it can be stored and later recovered. Sulphur has been suggested as a way for achieving this, either thermally, chemically, or electrochemically.



Fig. 1: Trough-shaped parabolic reflector.



Fig. 2: Parabolic dishes.

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hile the cost of renewable energy continues to fall as it achieves economies of scale, especially for wind and solar applications, there remain two basic problems with power from these sources. One is that it is not always most efficiently generated in the region where the power is required. The other is the essential variability of output from renewable sources, according to whether the sun is shining or the wind blowing. The latter in particular makes it impossible to use for 'base load' power generation, which to date still requires either a fossil fuel source or nuclear power. Attempts to overcome this issue often rely upon storing the renewable energy in a chemical form, such as by electrolysis of water to generate hydrogen and oxygen, or by using an insulated thermal medium to store radiant heat from solar capture.

Sulphur as a heat transfer fluid

Liquid sulphur has been investigated as a physical storage medium for solar energy as an alternative to molten salts, using sulphur as a heat transfer medium because of its lower melting point than traditional salts and high specific heat capacity. One such strand of research is being performed by Solar Research and Development Ltd, originally a Hungarian-based company but now relocated to the UK¹. Their chosen heat transfer medium *Sulfad*, is a mixture of sulphur with a small amount of catalyst.

Concentrating solar power (CSP) plants produce power by first converting solar energy into heat, next into mechanical power, and lastly, into electricity in a conventional generator. The three types of existing technologies involved are trough, dish/Stirling, and 'power tower' systems. Trough systems concentrate the sun's energy onto a receiver tube located along the focal line of a parabolically curved, trough-shaped reflector (Figure 1). Oil flowing through the receiver tube is heated to about 400°C; the heat is collected and used to generate electricity in a conventional steam Rankine cycle. Dish/Stir-

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Fig. 3: Power tower with heliostats.



Source: Solar Research & Development Ltd

Table 1: Comparison of energy storage densities

Technology	Energy density (kJ/kg)	Volumetric energy density (kJ/l)
Hydrogen	141,886	~6,700
Gasoline	47,357	~35,000
Sulphur	9,281	~18,000
Lithium ion battery	580	~730
Molten salt	282	~540
Elevated water dam	1	~1
Source: DLR		

ling systems focus the solar radiation at the focal point of a parabolic dish, which tracks the sun over the course of the day; temperatures reach about 750°C (Figure 2) but can go as high as 2,000°C. An engine/ generator located at the focal point of the dish converts the absorbed heat energy into electricity. The third type of technology, power towers, includes a field of heliostats that reflect the solar radiation to a receiver located on top of a tall, centrally located tower (Figure 3). The solar energy is absorbed by the molten-salt working fluid flowing through the receiver, which is located on top of the tower. Power towers provide for energy storage for up to several hours at 560-1,000°C in large tanks located at the base of the tower. When needed, hot salt is removed from the storage tank and used to generate electricity in a conventional steam turbine.

At the moment most research and development is focused on improving process efficiency and lowering investment and

ISSUE 378 SULPHUR SEPTEMBER-OCTOBER 2018 operational costs. One of the key questions is the nature of the heat transfer fluid. For example, while alkali metal nitrates like LiNO₃, NaNO₃, KNO₃ are considered to be promising candidates. They are expensive and remain sensitive to thermal decomposition. An ideal candidate for heat transfer fluid has to be stable at high temperatures, have a high thermal capacity, low vapour pressure and has to be in liquid phase at acceptable low temperatures. Solar Research and Development have focused on sulphur as it does not decompose in the same way as salts, has a relatively low melting point (120°C) and relatively low boiling point (444°C at atmospheric pressure). It is also less than one fifth of the cost of conventional salts. The aim of the company's research is to develop a sulphur based heat transfer system in either a parabolic trough or tower type, using the liquid/vapour phase to gain the most intensive heat transfer coefficients and lower volume/surface and pressure requirements. The boiling and superheating happens in the receiver section and condensation happens in the heat removal section (Figure 4).

At present, however, this technology remains in its infancy. The company is still attempting to develop a pilot plant at the SOLUCAR facilities near Seville in Spain to test the concept beyond the laboratory stage.

Chemical storage

Storing electrical energy as a chemical has been touted as a way of large scale storage of energy, especially in remote areas. Hydrogen, methane and even ammonia have been suggested, but there is also work being conducted on using sulphur as a chemical storage medium, as initially suggested by ASRL's Peter Clark and Norman Dowling in 2003². As Table 1 shows, while sulphur may not be as chemically efficient as fossil fuels, it can be far more efficient than battery or thermal/molten salt technologies. With \$1.8 million of funding from the US Department of Energy and \$900,000 of its own funding, General Atomics conducted work from 2010-2013 on supplying baseload power using a concentrating solar power (CSP) plant integrated with sulphurbased thermochemical heat storage³. The technology stores high temperature solar heat in the chemical bonds of elemental sulphur. Energy is recovered as high temperature heat upon sulphur combustion, while the solar heat itself is used to decompose sulphuric acid, allowing a route back

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for the sulphur dioxide generated by burning sulphur via a disproportionation reaction (Figure 5). Most of the experimental and developmental work was associated with the SO₂ disproportionation and sulphuric acid decomposition reactions. The former was studied using thermodynamic modelling and laboratory measurements, which showed that full disproportionation can only be achieved at elevated system pressure. A conversion rate of up to 30%/ hr was demonstrated, which far exceeded the original target of 10%/hr. Molten sulphur is separated from the acid product by means of their density difference.

As far as the sulphuric acid decomposition reaction was concerned, the study concluded that the directly irradiated decomposer by General Atomics and DLR is best suited to the current plant design. Following testing, a redesign for a receiverreactor in the 100-500 W range suitable for industrial conditions was carried out which uses a secondary concentrator to maximise radiation collection and provide rapid heating of process gas. Catalyst testing was also performed, with $CuAl_2O_4$ showing promise, but General Atomics said in its final report more long term testing of this and other catalyst candidates is needed. An overall storage cost of \$2/ kW-hth was determined. This very low storage cost makes the technology suitable for seasonal storage. A third phase of the project was however abandoned after the DoE and General Atomics were unable to agree on the attribution of costs.

PEGASUS

In Europe, work on sulphur as a chemical storage medium has been conducted with EU funding by the Karlsruhe Institute of Technology, the German Aerospace Centre (DLR) and the Centre for Research and Technology CERTH in (Greece). Industry partners include Brightsource Industries from Israel, Processi Innovativi, Italy, and Baltic Ceramics, Poland. The project – PEGASUS – is funded under the EU Horizon 2020 Framework Programme to the tune of €4.7 million.

DLR was one of the partners working with General Atomics on the DoE project, and PEGASUS is in many ways a continuation of the same principle, coupling solar renewable power generation with a sulphur storage cycle. The main difference is that the PEGASUS concept uses solid particles as the heat transfer fluid, which can also be used for direct thermal energy storage with indirect thermochemical storage of solar energy in solid sulphur, allowing round the clock renewable electricity production⁴. Like the General Atomics project, it uses a solar centrifugal particle receiver, sulphuric acid evaporator, sulphur trioxide decomposer and sulphur combustor. Elemental sulphur is produced by the disproportionation of sulphur dioxide, i.e. conversion of sulphur dioxide into sulphur and sulphuric acid. The focused sunlight of the solar power plant supplies the process heat with the energy and temperature required to close the sulphur cycle and to convert sulphuric acid back into sulphur dioxide in the presence of suitable catalysts (Figure 5).

Testing of the particle receiver is to be carried out in the high-flux solar simulator of the German Aerospace Centre (DLR) in Juelich, Germany, and ultimately it will be integrated with the evaporator and the decomposer to demonstrate the suitability of the concept. The Karlsruhe Institute of Technology section focuses on the technical implementation of combustion. It is planned to develop a sulphur burner for stable combustion in the range from 10-50 kW at high power densities under atmospheric conditions and temperatures higher than 1,400°C. Power density in particular allows for the effective use of sulphur as a fuel for electricity production.

A detailed flowsheet is planned to be drafted and the optimised integrated process scaled to the 5 MW thermal power level. Prototypes of the key components, such as the solar absorber, sulphuric acid evaporator, sulphur trioxide decomposer, and sulphur burner will be developed and tested at the solar power tower facility. In addition, the materials required for heat absorption, transfer, and storage and for the catalysts of the chemical reactions are planned to be tested for efficiency and longterm stability. The project runs to 2020.

Masdar

Finally, mention must be made of work being conducted in the United Arab Emirates (UAE). In a region where there is no shortage of sunlight and plenty of empty space in which to set up solar arrays, and, via Abu Dhabi state oil company Adnoc, one of the world's largest sulphur production capacities. Abu Dhabibased renewable energy company Masdar has been attempting to marry the two of these using a sulphur and sulphuric acidbased cycle driven by solar energy using the General Atomics concept, although Masdar is also investigating the possibility of additional hydrogen generation by using steam from the waste heat boiler of the sulphur burning plant to electrolyse water. The first phase of the planned road map, to take place over three years, will build a 50+kW demonstration plant using DLR's sulphuric acid decomposer, General Atomics' disproportionation reactor, and Masdar's own solar tower array in Abu Dhabi, and integrate them into a working solar sulphur cycle plant⁵. Masdar owns and operates the innovative Shams 1 facility, a 100 MW concentrated solar power (CSP) plant at Masdar City, which represents 10% of total global CSP capacity and 70% of Middle East CSP capacity.

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Battery storage

The 'holy grail' for electrical power generation would be large scale storage of electricity in a battery array, allowing power to be stored electrochemically. Sulphur may also have a role to play here. There have been experiments with large scale battery storage using lithium-ion batteries, and in 2017 large scale battery storage capacity reached 1GWh, with new large scale storage projects launched including the 120 MWh system in Escondido/ Aliso Canyon, California, and Tesla's 129 MWh battery in Hornsdale, South Australia. Although costs for large scale lithium ion battery systems has fallen by 80% in just a few years, it could still be possible to reduce weight and cost using alternative technologies. Much work has been conducted on lithium sulphur (Li-S) batteries, which have 2-3 times the capacity of Li-ion and a fraction of the cost, as we discussed in Sulphur 3616.

However, the practicality of Li-S technology is hindered by technical obstacles, such as short shelf and cycle life (less than 100 recharge cycles, compared to thousands for Li-ion) and low sulphur content/loading, arising from the shuttling of polysulphide intermediates between the cathode and anode and the poor electronic conductivity of sulphur and the discharge product Li_2S . Much progress has been made during the past few years to circumvent these problems by employing sulphur-carbon or sulphur-polymer composite cathodes, novel cell configurations, and lithium-metal anode stabilisation, but work clearly remains to be done⁷.

Many uncertainties ahead

This is an area where the technology is rapidly evolving, and predictions difficult to make. The money committed to the twin demonstration projects at Masdar in Abu Dhabi and PEGASUS in Germany appear to show that using sulphur as a chemical storage medium for solar energy is still in the running as a viable alternative to other storage methods. However, while the market for energy storage is predicted to increase seventeen-fold over the next five years, sulphur remains only one of many competing alternative technologies, and with the cost of batteries coming down it still remains an open question as to whether this can ever be a major use for sulphur or merely an interesting niche option.

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SUDIC 40 years on

Jerry D'Aquin of ConSul Inc looks at the SUDIC guidelines for producing formed sulphur 40 years on from their origins, and whether adjustments in the specification are warranted.

he Sulphur Development Institute of Canada (SUDIC) produced its recommended guidelines for the production of formed (i.e.: produced through a manufacturing process) sulphur in a definitive 1977 report. The pioneering work commenced in 1976, conducted by the institute's Sulphur Industry Forming Committee. It was to "identify methods by which an improved form of export sulphur could be developed for shipment to the export market". The 1977 SUDIC guidelines recommended product characteristics likely to improve product quality by minimising airborne emissions of sulphur during handling and transport from Alberta to global customers remain largely valid forty years after publication.

This pioneering work, conducted by the Canadian sulphur industry's leading experts, provided the guidelines necessary for developing improved processes and materials. We are most appreciative of their hard and prescient work. However, minor adjustments, benefiting from 40 years of production and handling formed sulphur, are now recommended. Most adjustments are linked to optimistic laboratory assumptions and, as in the case of H₂S content, new standards refined by operating results during commercial operation.

The guidelines and their rationale were based on extensive research and testing by SUDIC, then the world's leading sulphur research entity, and its contractors. In total SUDIC evaluated 11 technologies

Property	Standard specification	Premium specification
Shape/size:		
Generally spherical	2-5 mm diameter	2-4 mm diameter
		75% between +/- 1 mm of average diameter
Size distribution:	>6.3 mm: <5%	>4.75 mm: <5%
	<1.18 mm: <2%	<1.18 mm: <2%
	<0.3 mm: <0.25%	<0.3 mm: <0.1%
Friability:		
Stress level I (abrasion)	<2% fines (<0.3 mm)	<1% fines (<0.3 mm)
Stress level II (impact)	<5% fines (<0.3 mm)	<0.5% fines (<0.3 mm)
Bulk density:		
Loose	>1,040 kg/m ³	>1,040 kg/m ³
Compacted	>1,200 kg/m ³	>1,200 kg/m ³
Compaction breakdown:		
(25psi, equiv to 15 m stockpile)	<0.5% fines (<0.3 mm)	<0.2% fines (<0.3 mm)
Angle of repose:	>25°	>25°
Moisture content:	<1.5%	<0.5%

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for forming molten sulphur, along with preferred product shape, size, size distribution and testing protocols to provide common ground and guidance for equipment manufacturers developing formed sulphur products. The guidelines recommended that the end-product should be generally spherical, smooth, free of large particles and of low friability. It should also create little dust during handling and transit. Holding all sulphur shipped from Vancouver to such standards would, it was hoped, confirm Canada's position as the global leader in sulphur technology and garner higher values for Vancouver exports¹.

The SUDIC guidelines were proposed forty years ago, long before modern forming technologies developed (as a consequence of these recommendations) and became operational. The guidelines' publication also precedes many current safety, health and environmental (SHE) practices. Richard Hands' paper on Sulphur Quality Specifications in *Sulphur* 356² notes several issues which need to be addressed. Two obvious ones are: specifications for formed sulphur "as loaded " to ship; and the product quality and preventing SHE incidents.

The author has travelled to the Arab Gulf since 2010 and is aware of "pops", dust, fires and explosions related to sulphur handling in the Middle East. These may be linked to misinterpretation of, or omissions in, the guidelines. Some are similar to events at Vancouver's two terminals prior to the installation of additional water/foaming applications. Foremost, when evaluating guidelines with the benefit of hindsight, is to underscore that the SUDIC report was intended to provide guidelines for equipment manufacturers seeking to develop improved formed sulphur products.

Forty years later, operating experience, technology and materials handling enhancements warrant a second look at SUDIC's recommendations. The article make extensive use of the SUDIC guideline report of June 1977 and other documents, articles and speeches by authors of that seminal work, personal study supplemented by visits and work at forming and receiving facilities in North America, Europe, North Africa, Asia and the Arab Gulf.

Table 1 provides a list of SUDIC premium and standard formed sulphur guidelines as published in 1977.

Figure 1 shows a grab sample form the Strachan, Alberta, Polish (air) Prill forming

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Fig. 1: Polish air prills.

facility. That unit is in the process of being deactivated due to reduced volumes and pollution. The air prill process has not been available for sale since the 1980s due to a catastrophic explosion and airborne pollution. Note the even, spherical particles, polished surfaces and small size (1-2 mm): truly a premium product from which the foregoing guidelines were derived.

The intervening years

Changes since SUDIC's report was issued in 1977 should be kept in mind. Firstly, SUDIC is no longer in existence, although some of its functions have been partially assumed by Alberta Sulphur Research Ltd (ASRL) due to organisational and funding changes. Secondly SUDIC's preferred formed sulphur process, the 'Polish prill', upon which its premium product guidelines were based is no longer commercially available as a consequence of irresolvable pollution flaws and explosion risks. Western Canadian sulphur producers also did not invest in covered storage, enclosed railcars or moisture-free transfer procedures to deliver sub-0.5% moisture formed sulphur to ships' holds at Vancouver as the report expected.

Many forming facilities have been built in the US and Arab Gulf specifically in the past decades. These provide realtime operating information (rather than assumptions) with which to examine key technical and commercial issues surrounding 'dry' versus 'wet' formed processes. It is demonstrable that the handling and transport of formed sulphur carrying <0.5% moisture results in a portion of the sub-300 micron sulphur particles becoming airborne causing hazardous volatile sulphur particulate (VSP) conditions; the trail transit from Al Hosn and unloading at the Ruwais terminal in Abu Dhabi is an example. Conversely, 'wet formed' sulphur manufactured by immersion in water is a globally traded material incurring no qual-

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ity discount. It is preferred by some users as moisture limits VSP emissions. Spraying water or water-surfactant mixes is also an approved method to reduce VSP emissions at leading load ports and dis-ports from Vancouver to Africa.

Reasons for revisiting SUDIC

- 1. It is now possible to update the report with knowledge from forty years of design and operating experience. I have seen from personal communications and photographs that dust, fires and explosions occur at Adnoc. Kuwait and Qatar sulphur forming and shipping operations as they strive to handle sub-0.5% moisture sulphur following production. Limiting moisture to less than 0.5% in an effort to meet the premium product guidelines determined by SUDIC is based on a defunct and dangerous forming process and untested dust control assumptions, and contrary to modern SHE practice. Volatile sulphur particulate (VSP) emissions from 'dry' sulphur causes 'pops' (minor static electricity discharges), fires, large explosions and unhealthy worker conditions. Revising the moisture guideline would reduce VSP and its consequential risks.
- 2. One of SUDIC's goals was to reduce moisture impact on Canadian sulphur transport costs. Sulphur producers determined that the added costs to deliver dry sulphur to ships (via covered storage) was excessive compared to freight savings and never followed through. Vaporising moisture in the range of 1-3% is not really a cost for modern sulphuric acid plants, as these generate sufficient excess low pressure steam to melt 0-3% moisture for no incremental cost³.
- 3. The guidelines were for 'as produced' sulphur. Surprisingly, no detailed industry standard exists for particle size distribution or moisture of formed sulphur loaded to ship. The need is now urgent as the supply of formed sulphur increases and production has migrated from North America, where 'Vancouver' set an unofficial standard. While that may have been reasonable it becomes risky and very costly as load ports multiply and experience fails. The only adjustment accepted in most contracts for sulphur loaded to ship relates to normalising Bill of Lading weight to 0.5% moisture in order to elimi-

ISSUE 378 SULPHUR SEPTEMBER-OCTOBER 2018 nate the impact of excess moisture on billable tons.

- 4. It is necessary to implement procedures to prevent cargo contamination by iron and seawater.
- Maximum H₂S content in both molten feedstock and formed product should be contractually defined. A 30ppm level is recommended for all forms of sulphur unless local standards are stricter. It improves friability and SHE.
- 6. Sulphur particulates smaller than 300 micron/50 mesh are potentially deadly. Appropriate SHE procedures, including VSP mitigation and effective operator inhalation protection, must be enforced. In order to achieve this, the moisture content of formed sulphur being transferred immediately following production should not be less than 1%. Minimum moisture of not less than 1.5% is recommended during vessel loading, with 2-2.5% being acceptable. Certain load ports may require additional moisture. Sulphur sampling procedures should measure moisture as the cargo enters ships' holds. Moisture determination must include all water used independently or in combination with other products (surfactants for example) from the sampling point and to its exit from the shiploader's spout in order to properly adjust the Bill of Lading.

The guidelines – a review

The following paragraphs review SUDIC formed sulphur guidelines, and introduce new considerations beyond those embodied in that report, pointing to suggested updates for seller and buyers to debate. We must again remember the guidelines focused on "as produced" characteristics and omit the phrase "as loaded." The latter was beyond SUDIC's remit since no modern forming process existed in Vancouver and the Polish process only operated in Poland.

Since formed sulphur is now produced and loaded throughout the globe for use thousands of miles away, load port quality standards are increasingly needed. I once was called to Odessa, Ukraine, to examine retention samples from a sulphur cargo rejected for loading by the ship's Captain for being "out of specification." The error was easily corrected when opposing experts viewed the material and readily agreed to its manufacturing provenance. Industry-accepted specifications would have easily provided guidance and con-

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tract clarity rather than requiring an "expert opinion". Such could be a task contracted to The Sulphur Institute or others.

Preferred Product

SUDIC's "Preferred Product" was the Polish Prill (Figure 1). This type of material is perfectly spherical, small in diameter, smooth-surfaced, resistant to breakage and easy-flowing. Polish Prill sulphur is truly a premium formed sulphur material and no other material we have seen matches its characteristics. Unfortunately, deployment showed two fatal flaws. The first was severe pollution from sulphur dust exiting the top of the tower; leading to extreme downwind soil acidification. Apparently, airborne dust was not a problem in communist Poland where the technology was developed. The second setback was a devastating explosion in Jubail, Saudi Arabia, which consumed two prilling towers and a 70,000 tonne storage building in 1985. The explosion arose from the combination of extreme temperature, humidity and operating parameters required to achieve the desired production rates in that climate. Static electricity build-up in sulphur particles while swirling in the tower apparently led to sparking, causing sulphur dust to ignite, triggering a conflagration and the subsequent demise of Polish Prill technology. In truth, sulphur particle emissions from similar facilities in Alberta have caused irreplaceable ecological damage through acidification.

Environmental considerations

SUDIC guidelines were proposed to help technologists to develop equipment able to produce formed sulphur material with lower dust emissions in order to reduce the environmental impacts of the material's transit in Alberta, British Columbia, Vancouver and at global destinations. SUDIC's emphasis was to limit the creation of tiny particles during transit by selecting the least friable material. It seemed to lack a focus on defining the type of particulate which caused visible dust floating in the atmosphere at transfer and unloading points. The authors were also intent on finding a solution that would not include moisture, i.e. a 'dry forming' method in order to reduce transport costs. However, several comments regarding dust minimisation such as the following can be found in the report¹:

"Whether a dust problem will be encountered in bulk movement of even these products is not known at this time."

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"It is expected that related dusting will be below acceptable levels but this must be verified by trial shipments."

"The requirement of a 1% moisture level to minimise pellet dusting has not been demonstrated."

Geographic considerations

The report states that climatic conditions in various parts of the globe might require adjustment of moisture guidelines. To our knowledge from industry contacts and operating observations, such operating adjustments are frequent in Canada (e.g. spraying storage piles in summer, adding moisture at loading and in the terminal) where operational experience is well-established. Unfortunately this is not the case at Middle East installations where HSE problems have occurred due to an inflexible reverence for the SUDIC guidelines.

"Thus it is strongly recommended that bulk shipment of dry product be moved through the West Coast terminal handling facilities with careful attention to dusting, conveyor performance and storage aspects."

Other objectives

While SUDIC focused on developing product guidelines leading to less dusty formed sulphur, it concurrently sought to also promote Canadian formed sulphur technology and reduce transport costs². The first objective, for Canada to be seen as the source of "leading sulphur technology" was achieved when a Canadian company obtained the license to market 'Polish Prill' technology. It was then modified, used in Canada and promoted globally as SUDIC's recommendation for making premium formed sulphur.

SUDIC also sought to reduce sulphur transport costs by limiting moisture to unprecedented levels while concurrently reducing VSP emission. Moisture weight causes higher rail and marine transport costs per net tonne of sulphur. Lower rail charges to Vancouver meant higher producer returns. SUDIC's objective was thus to encourage dry forming processes. The report's authors assumed under 0.5% moisture would be sufficient to reduce airborne particulate dispersion to acceptable levels (though they did note that in certain climatic conditions more water might be needed - see above). This may have been possible with Polish prills if all sulphur came from such a source, but it has not proven feasible in this writer's experience.

Low-moisture (< 0.5% at Vancouver) proved impossible to deliver at dockside for lack of infrastructure investment. Sulphur producers and transport company Sultran determined the estimated savings from transporting sulphur with minimal water content did not justify the capital investment in covered storage at both the point of origin and destination plus the use of covered rail cars. Complex operating procedures would also be required to maintain such low moisture, including halts to vessel loading during inclement weather.

In contrast, many Arab Gulf producers strive to maintain their formed sulphur below 0.5% moisture level, believing consumers will always prefer zero moisture material. This position ignores that all sulphur from North America is loaded at moisture levels exceeding 1.5% and that lack of moisture has led to events which have damaged plant and equipment.

Although the 0.5% moisture guideline remained unachievable at Vancouver, it became entrenched in sales contract specifications. All formed sulphur sold in bulk in North America is assumed to include at least 0.5% moisture. In other words, a US\$100/t Vancouver spot sale price reported by trade publications is assumed to include 50 kg of water. The actual price of that sulphur, dry basis, is effectively US\$100.50. Commercial transactions use the actual moisture of the cargo as determined from samples loaded to vessels and reduce tonnes loaded by the moisture contained, except for that one half of a percent (0.5%). The resulting amount represents the commercial sulphur tonnage listed in Bill of Lading for invoice purposes.

Marketing considerations and the demise of SUDIC kept the 'premium' sulphur appellation in use in spite of the inability to deliver that quality to ship holds at Vancouver. Once established, production guidelines were never revisited or expanded to more essential "as-loaded" specifications. Producers and terminals operators simply applied moisture as and where needed to achieve safe and environmentally required loading operations.

Buyers were equally remiss. They never challenged why significantly higher moisture was always loaded into their ships when "Premium" sulphur was purchased at 0.5% moisture. Based on SUDIC's guidelines buyers who purchased "Premium" sulphur should have requested a marine freight discount for "excess water".

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Fig. 2: Sulphur granules.



Fig. 3: Sulphur pellets.

Fig. 4: Sulphur pastilles.

Fig. 6: Particle sizes of three formed sulphur types



Moisture

The inescapable fact of formed sulphur transport and handling is that moisture is essential to mitigate VSP and sulphur dust. *Sulphur*'s article on forming in issue 356² notes that in a test " three samples of premium formed sulphur were transported with particle sizes, fines and moisture measured at loading and discharge. All three samples generated the same amount of fines during transit." However: "... the actual quantity of dust observed during unloading was effectively zero for the sample with the higher moisture content, but clearly observable for the one with the lowest."

The article continued with the following question: "given that there is evidently a trade-off between actual observable dust and moisture content, should we always necessarily be seeking the lowest possible moisture content?"

The question is not rhetorical and must be addressed. New sulphur forming installations are cropping up annually in the Middle East, with the majority adhering to the "less than 0.5% moisture" guideline. These 'dry' materials have SHE impacts wherever formed sulphur is produced, shipped, received and utilised. Eye and lung irritation/disease and dispersion of VSP in the environment, explosions and fires would be significantly reduced by adding some moisture to formed sulphur.

Much has been learned by society and industry regarding airborne contaminants over the past forty years, including organisations' greater responsibility towards the environment, society and employees. It is time for all segments of the sulphur industry to recognise the significant benefits a small amount of water/surfactant can bring to lowering volatile sulphur particulate (VSP) emissions and enhancing safety, health and environmental benefits.

Modern formed sulphur

Current manufacturing technologies produce solid sulphur in three general shapes: granules, pellets and pastilles. Figures 2-4 show these well-defined shapes.

Each shape is the result of a specific forming process/technology pursued pri-

marily by three separate companies while keeping the SUDIC guidelines in mind: Enersul, the former Devco (now Matrix PDM) and the former Sandvik (now IPCO). Each technology produces excellent material for shipped sales of formed sulphur in bulk. However, each also has characteristics which create a source of particles which, during transport from the outlet of each forming unit to point of consumption, fit the size and shape profile of volatile sulphur particulate (VSP). For example, granules, by the very nature of an accretive production process, may be spherical but have a rough surface. This gets polished during transport by rail or truck, creating very small particulate which easily becomes airborne at transfer points. Such is evident when unloading sulphur from rail cars at the Ruwais, UAE, terminal. Pellets have uneven surfaces and cavities which can break and erode during transit, causing the volume of small pieces in a sample to increase, and finally pastilles have a flat bottom, rounded top, smooth surfaces and sharp edges. It resembles the yolk (yellow part) of an egg when it is set on a flat sur-

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face. As pastilles tumble through transfer points, edges tend to chip off and erode, providing a source of particle degradation.

Vancouver's sulphur operating practices provide unquestionable proof to even the most adamant sceptic that VSP is generated during rail transit. Pacific Coast Terminals and Kinder Morgan's sulphur terminals, the only operating transfer terminals in the area both unload sulphur railcars using rotary dump systems equipped with copious spraying of water/surfactant. Both use this approach to capture VSP and preclude environmental violations, neighbour complaints and the risk of fire/explosion. An initial application of moisture is usually made at most forming plants in Alberta.

Size

Figure 5 is a size distribution chart for each type of formed product obtained from screening samples taken at the indicated manufacturing location. Two are in Alberta and the third in California. Figure 6 also provides size dispersion as unloaded at destination. These samples were not collected from the same production batches as the preceding dispersion chart; they are meant to provide a general comparison of degradation during transport.

Pastille/Sandvik size dispersion was 68% via a #8 Tyler Mesh Screen or 2.38 mm. At least 2.5% of the sample was below 50 mesh (0.3 mm or 300 microns). Sub-300 micron particles contain VSP. The total material of 300 microns or less is 4% of the screened sample, which is very high compared to SUDIC estimates.

Granule/Adnoc size dispersion was 37% via a #6 Tyler Mesh Screen or 3.36 mm. At least 1.5% was at, and 0.7% below, 50 mesh (0.3 mm/300 microns). The total sample was found to contain 2.2% of particles 300 micron or smaller. This slightly exceeds SUDIC's recommended highest results for Stress Test II. From a practical standpoint, while this is the least amount of sub-300 micron particulate found among the three types of formed sulphur, this specific material causes dust-related issues at load and discharge ports.

Wet Prill/Pellet size dispersion was 63% via #6 Tyler Mesh Screen or 3.36 mm. At least 1.7% was below 50 mesh. Total particles of 300 micron size or below was about 2.5%. This material has the lowest VSP emissions.

On the basis of this very preliminary and primitive work, if half of the sub-300 micron material was lost at unloading and



Fig. 7: Sub-300 micron sulphur retained from two samples from Ukraine.



Fig. 8: Electron microscope pictures of sulphur particulate.

transfer points, it would represent between 500 tonnes (half of $2.5\% \times 40,000$ t) and 140 tonnes (half $0.7\% \times 40,000$ t). With shipments of 1 million tons per year, the loss would rise to between 3,500-12,500 tonnes, worth up to \$1.25 million.

Reclaim and handling

This is where external physical forces start affecting even the hardest form of sulphur which technology can produce. Any formed sulphur product can split or shatter when striking hard surfaces, such as steel bulkheads or hopper walls at conveyor transfer points. Start-ups are particularly harmful as newly formed sulphur falls long distances without the cushioning effect of a pile of previously existing cargo. Drops into empty hoppers at transfer points or storage bins can easily split large particles. Then comes degradation during reclamation which uses drag paddles or front end loaders to pull, push or scoop formed sulphur to ship-loading conveyors.

The tyres of large loading equipment pulverise granules, pellets, or pastilles at the forming plant, and during ship loading and unloading as vehicles travel across hard storage pads or ship hold surfaces with sulphur underfoot.

Reclamation and loading causes more degradation in retrieval, transfer points and when product is dropped into ship's holds. Initial loading into each of the

ship's five holds can free-fall as much as fifteen metres onto flat steel plates. easily breaking the material regardless of its quality. The loader operator's incentive is to accelerate conveyor speed and moisture addition to cover VSP from this aggressive loading process. To mitigate small explosive 'pops', Vancouver operators add moisture through sprays from nozzles attached to the end of loading spouts. Some loading ports or terminals use fire hoses to apply water directly to conveyors and the holds in an effort to limit VSP, but it leads to an additional, un-measured, volume of water which is counted as sulphur being sold.

As a consequence of handling alone, the quality of material loaded to ship and consumed in buyers' melters can vary significantly from production samples. The photographs in Figure 7 show the amount of sub-300 micron sulphur present in two granular sulphur samples loaded to the same vessel at a port near Odessa, Ukraine. It is an amazing contrast between samples which were collected and processed in accordance with local and international practices. If the sulphur industry had "as loaded specifications" with particle size requirements one of the samples would not have passed.

Volatile sulphur particulate arises primarily from sub-300 micron particles of sulphur. Con-Sul believes dry particles below

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150 microns easily become airborne as sulphur is transferred.

Figure 8 shows two electron microscope photos of sulphur particulate. Each has a line on the bottom showing relative size. The one on the left is 500 microns (0.5 mm) long. The photo on the right is taken at a much greater resolution with the line representing only 10 microns. An interesting characteristic of these particles is their relative length vs. depth. They might be equated to broken pieces of glass. This wide surface area in relation to mass is the determining characteristic separating VSP from non-volatile material. The latter simply have too small of a surface area to mass ratio. This same physical characteristic allows us to examine control of VSP in a new light, visualising surface tension along the wide areas of these microparticles as a key binding characteristic.

Moisture

Most sub 300 micron (50 mesh) particles in sulphur cargos are potential VSP. These can be bound into larger, non-volatile larger particulate through moisture's surface tension on their flat surfaces. SUDIC guidelines advocated against the use of moisture to reduce transport and melting costs, but those points were discussed and mitigated earlier. The lack of market for moist sulphur was also debunked with the fact that all formed sulphur exported from North America is shipped with at least 1.5% moisture and sometimes as much as 4% with no discount other than normalising sulphur weight loaded to 0.5% moisture.

The unequivocal proof that moisture is needed for safe transfer of formed sulphur is that it has been applied for decades to sulphur shipped from Vancouver's Kinder Morgan and Pacific Coast Terminals to all parts of the world. Forming which takes place in British Columbia uses Enersul's WetPrill forming technology. Solid sulphur (slate, granule and prills) from Alberta, much of it produced with less than 0.5% moisture, is exposed to precipitation and receives moisture when loaded to rail cars. The cargo is unloaded at Vancouver's terminals using rotary dumps systems with dust-suppressing water-based sprays in use, stored in open-air piles (water is at times sprayed when a pile's surface dries out), placed on ship-loading conveyors with front-end bucket equipment which scoops sulphur from moist or wet storage surfaces (to preclude sparks with powdered sulphur present). Dust emission during ship loading is mitigated using water-based foams.

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Tiny volatile sulphur particulates are created during transit from Alberta and handling at the terminal. Front-end loader tyres crushing granules as they load conveyors are particularly bothersome. Even the hardest formed sulphur particles are inevitably pulverised. By the time formed sulphur is loaded to ship at Vancouver moisture analysis can range from 0.8% to over 3% and sampling on-board ship has shown 300 micron material to reach 3%. Moisture added for transit and storage, plus precipitation, precludes most VSP from being released in transit, leading to greatly reduced airborne pollution, fires and explosion. Had the SUDIC Formed Sulphur Product Guidelines Task Force continued in place into the 1980s, they would likely have revised the 0.5% moisture guideline as being insufficient to preclude hazardous dusting when formed sulphur (other than perhaps Polish Prill) was transported under carefully controlled conditions.

In an interesting technology transfer, SUDIC's 'low moisture' message has found strong advocates in the Arab Gulf and at some Middle Eastern sulphur forming facilities. Most marketing organisations in that region, except for Aramco, firmly believe formed sulphur with more than "0.5% moisture when loaded to ship" is an unsatisfactory commercial product. Arab Gulf marketers believe buyers will avoid such sulphur (despite proof to the contrary in North America) unless no other material is available. The SUDIC moisture guideline is diligently monitored. As a consequence, most installations in the region experience explosions, fires, 'dust' incidents and related emergencies plus uncontrolled emergency 'dousing' with water from fire hoses. Recipients of this material are known to suffer fires, explosions, VSP emissions and other SHE concerns, including employee health hazards.

Corrosion

One item which prevails in the industry is the alleged direct link between sulphur, moisture and the creation of highly corrosive sulphuric acid. The assertion has always been confusing, as water and sulphur placed together in a glass beaker remains inert – that liquid does not corrode. Moisture with fine sulphur particulate will create an environment which specific acidifying bacteria can colonise and generate sulphuric acid. Experience and tests have shown such corrosion to be minimal during vessel transits.

The real cause of severe corrosion on ships and port equipment related to sulphur is an electrochemical reaction which occurs when sulphur, moisture (even in small quantities) and iron or steel come into direct contact with one another. That reaction is described at length by ASRL in past research and most recently in a paper entitled Corrosion Due to Elemental Sulphur in Sour Gas Production and Claus Recovery Systems by Drs Peter Clark and Norman Dowling associated with Alberta Sulphur Research Ltd. The simplest, most effective solution for this type of problem is to maintain separation and preclude any sulphur-steel contact. Simply put, to avoid sulphur-steel corrosion: keep it painted! Unfortunately, such basic advice has been significantly diluted over time for minuscule cost savings which can cause significant harm to the sulphur cargo.

Reconsidering sulphur specifications

Shape

Different types of modern formed sulphur products have been developed since the SUDIC guideline report. Three companies' products have reached global industry acceptance. Slight modification of the SUDIC guidelines is warranted to bring reality, clarity, practicality and agreement to industry communications: pastilles, granules and pellets, or other similar forms agreed by the sulphur producing and consuming industry, need to be acceptably defined.

A suggestion is: "pastilles, granules, pellets and prills produced in accordance with process vendor's instructions to meet IPCO/Sandvik, Enersul or DEVCO/Matrix PDM forming technologies specifications, plus others forms and processes as may be acceptable to buyer."

Size

SUDIC notes that large pieces of sulphur gather more momentum during transfers. That characteristic increases the risk of particle breakage during transfer. Broken edges are sharp, creating more the probability that tiny particles will be created during handling. Further research by ConSul in past years point to the guidelines' lower size specification needing to be extended below the 0.3 mm (300 micron) SUDIC reference level. Particles between 250 and 50 microns appear particularly apt to become and remain airborne. We note that further research is required in this area in order to improve VSP management practices.

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The ship's sample should reflect the following minimum requirements prior to or following loading. Sample shall not be handled or prepared in any way between production and testing to avoid degradation or elimination of small particles.

Particles above 3 cm = nil; particles below 3 cm to 2 cm = 10% maximum; particles below 2 cm to 1 mm = 80% minimum; particles below 1 mm to 0.3 mm = 5% maximum; particles below 0.3 mm = 5% max.

No visible opacity liberated from a 2 metre high drop-test stream with 5 km/h crosswind coming through a 30 cm x 20 cm opening (specifics of test to be determined and not to supersede local environmental regulations).

Moisture

Despite the general comments in its report, SUDIC's authors recognised their opinions "assumed dry sulphur could be transported without creating volatile dust." This paraphrased statement is found in numerous places in the report. Several references are made regarding the possible need to adjust moisture recommendations in certain climate areas.

It is now unequivocally accepted that moisture is an essential component to reducing VSP during transfers of formed sulphur. ConSul tests indicate that 2% moisture in the sample virtually eliminates sulphur particulate material from passing through a 300 micron screen. SUDIC specifications for 'Standard' formed sulphur calls for a maximum moisture content of 1.5% maximum. Our recommendation is for 1.5-2.5% moisture, evenly distributed, to enhance VSP mitigations.

Hydrogen sulphide content

This hazardous and explosive gas should be included in formed sulphur guidelines to conform with modern SHE practices. Many forming installations form un-degassed sulphur and conduct no monitoring. Molten sulphur feedstock being sent to forming units should not to exceed 50 ppmw when sampling at the inlet of the forming unit. Formed sulphur H_2S content should not exceed 25 ppmw when loaded to ship.

Ships' hold preparation and operating practices

Though not related to formed sulphur quality at loading, proper preparation of ships' holds to receive solid sulphur is essential to its proper quality when delivered to buyers. It also can be a critical component in achieving employee safety and fires associated with FeS. Full lime coating of exposed steel up the cargo's estimated loading mark should be mandatory unless another means is determined to be equally effective, at which point it may also be used.

Three times per day readings of bilge water levels should be required, as well as daily or more frequent pumping. Under no circumstances should water be allowed to stagnate on hold bottoms. Nor should bilge water be allowed to encroach above hold bottoms. Records of readings and pumping will be delivered upon arrival.

Conclusion

The foregoing is provided for discussion among industry members in an attempt to address product quality, uniformity, transactional accuracy and avoid communication problems which exist in the formed sulphur industry.

References

- 1. SUDIC Guideline Report, June 1977, Executive Summary, pp. i-xxi.
- 2. Sulphur 356, Jan-Feb 2015, pp24-29.
- 3. Discussion at Sulphur 2016 Conference (London) sulphur handling workshop.



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European sulphuric acid exports

Europe's sulphuric acid industry remains a major exporter of acid to other regions, but has seen a shift in the location of those exports as some markets contract and others expand.



urope is one of the world's major sulphuric acid producers and exporters, accounting for about 10% of global acid production, with a long legacy of industrial production dating back to the industrial revolution. Although acid plants in some countries, like the UK, have mainly closed down as traditional heavy industries found it hard to compete with imports from the rest of the world, other traditional producers still maintain large capacities. Both production and demand is relatively mature, with a fairly heavy concentration on smelter acid production, which provides the bulk of exports.

Sulphur production

About half of Europe's sulphuric acid capacity is based on sulphur burning and hence relies upon a supply of sulphur for its feedstock. European sulphur output runs at 3.7 million t/a, mainly from refining.

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European sour gas processing has been in long term decline, and the Lacq gas field in France, which was where many of the techniques for recovering and processing sour gas were developed by Total, and which became a mainstay of French gas production from the 1950s, ended commercial production in 2013. Likewise Wintershall's Staffhorst field in Germany, which has been producing sour gas for 40 years, is in long term decline. There is still production from, for example, ExxonMobil's Grossekneten gas field in Germany, where the H₂S content of the gas is up to 35% and sulphur output is at 800,000 t/a. Overall, just over 20% of Europe's sulphur production currently comes from sour gas processing.

On the refinery side, European demand for refined fuels is mature and in long-term decline. Europe's refineries have been gradually coming to terms with an imbalance in fuel production and demand in the continent,

whereby diesel demand has grown - forcing Europe to import diesel from Russia and other eastern states, while Europe has a structural surplus of gasoline, leading to exports mainly to the US. Europe's refining capacity is also in long term decline. The EU (plus EEA) has 85 remaining refineries with a total refining capacity of about 14.5 million bbl/d. This has fallen by a net 2.1 million bbl/d since 2000. As refining capacity shifts towards Asia and the Middle East, so European refineries will continue to face increasing international competition, especially for coastal refineries in northern and southern Europe - 'inland' refineries along the Rhine and Danube river systems are likely to be less exposed. It is likely that Europe will see further refining closures over the coming years.

This does not necessarily mean a decline in sulphur output, however. New IMO restrictions on sulphur content of marine fuels and a consequent decline in heavy oil demand mean that many European refineries are installing systems to upgrade heavy fuel oil, although many of these are delayed coking systems where the sulphur ends up on the petroleum coke rather than being recovered as elemental sulphur. There is also an incentive to deal with cheaper, heavier and sourer crude feeds. Overall, Integer Research predicts that European sulphur output is likely to remain fairly steady out to 2025.

Europe is a net exporter of sulphur and seems likely to remain so, but while Europe's exports of sulphur have often been as liquid cargoes, as with Canada, future access to international markets is likely to mean switching towards formed sulphur production. It is notable that Oxbow installed a sulphur former at Southampton, UK in 2014 to convert liquid sulphur from the nearby Fawley refinery into prills.

Acid production

Western European sulphuric acid production in the EU/EEA was 15.7 million t/a in 2016. Of this, the largest producer is Germany, with just over 4 million t/a produced that year, followed by Belgium, Spain and Finland, each with about 2 million t/a of production, and Italy with 1 million t/a. These five countries between them represent about 75% of European acid production.

Production comes mainly from sulphurburning acid plants (just under 50%), with smelters responsible for just under 40% of production. There is also some regenerated acid reclamation which accounts for 1.4 mil-

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lion t/a (9%) of production, and some pyrites roasting in Finland which generates another 600,000 t/a. However, two major smelters – Aurubis in Hamburg and Atlantic Copper at Huelva, Spain, as well as Boliden's two smelters in Skellefteham, Sweden and Harjavalta, Finland represent a major slice of European acid capacity. The sulphur-burning plants, conversely, are concentrated in Germany, Belgium, Poland and Italy, which represent half of all sulphur burning capacity between them.

Again environmental regulations may force a slight increase in acid production from metallurgical sources. There are also some ongoing improvement works. For example. Swedish mining company Boliden has contracted Outotec to design and deliver a new absorption section for its sulphuric acid plant at the Rönnskär smelter. Work will be completed in the second half of 2019.

Demand

While elsewhere in the world sulphuric acid consumption is mainly for fertilizer production, only around 20% of Europe's production goes towards this end use, with 60%

Europe's demand for sulphuric acid represented by chemical production, especially titanium dioxide production. There are broadly speaking two main production routes towards titanium dioxide - via a chloride route, pioneered by US companies and a sulphate route. The sulphate route is the older and has been historically regarded as more polluting, but European producers have developed far cleaner systems which recycle spent acid. Other chemical uses include production of other acids, as well as fibre manufacture, methyl methacrylate and aluminium sulphate. Non-chemical industrial uses make up most of the remaining 20% of demand, such as metal treatment - leaching, steel pickling etc. European demand for acid is largely mature, and although there are ups and downs due to the cyclical nature of many of the industries, there is no largescale growth.

Exports

European acid production tends to run in excess of acid demand, to the tune of about 3 million t/a. In 2017, Morocco was the largest destination for European acid,

at 950,000 tonnes, and this figure continues to rise as OCP continues to expand its phosphate processing capacity at Jorf Lasfar and other sites. OCP imported a total of 1.5 million tonnes of acid in 2017, and the figure for 2018 is expected to be close to 2 million t/a. Morocco's increased need for sulphuric acid has been something of a lifeline for European acid exporters because of declining demand in other regions. Cube once absorbed over 400,000 t/a of Europe's acid exports, but the installation of a sulphur burning acid plant at the Moa Bay nickel plant has seen this fall from 430.000 t/a in 2015 to virtually nil now. Brazil likewise is importing smaller volumes of acid, probably 200,000 t/a less in 2018 than in 2017, as producers of single superphosphate struggle against cheaper alternatives. There is a risk that - if sulphuric acid prices increase, as they have done this year due to outages at Japanese and Korean smelters, OCP may well switch back towards burning more sulphur domestically and importing less acid. Global phosphate and copper leaching projects have the potential to increase longer term demand, but not in the immediate future.





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Sulphur 2018 welcomes you to **Gothenburg**

TIL

CRU's annual Sulphur + Sulphuric Acid conference and exhibition will take place this year 5-8 November 2018 at Gothia Towers Gothenburg, Sweden. Attendees will have the opportunity to network with industry peers, attend workshops, hear the latest market analysis and price trends, take part in panel discussions and gain information from 36 technical papers covering operations, new technology, and equipment.

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ow in its 34th year, Sulphur 2018 is a premier industry event for business and professional development and high-level networking, regularly attracting over 550 delegates. Market presentations will feature CRU's global outlook for sulphur and sulphuric supply and demand and price trends and CEFIC's view on challenges and future opportunities for the European chemical market. In addition, there will be three panel discussions by industry experts to examine: IMO 2020 and how the industry is responding as the deadline nears; digitalisation/Industry 4.0 and the opportunities offered by digital transformation; the evolving energy mix and what it means for the sulphur and sulphuric acid industry.

The technical programme will feature a mix of new technology, process, equipment and material innovations, in addition to papers based on case studies focusing on operational experience and solutions to operational challenges. Technical sessions for sulphur and sulphuric acid will be run in parallel and will cover the following topics:

- Effective SRU operations: Managing your plant for optimum reliability and performance
- New plant and project execution case studies
- Tail gas treating and emissions management strategies
- Operational case studies: Maintaining and improving operations
- Emissions management and sustainable sulphur recovery
- Plant design options for improved safety and performance
- Sulphur operations troubleshooting clinic
- Effective temperature management for increased reliability and energy efficiency
- Acid gas and oxygen enrichment options
- New technology and equipment for increased efficiency
- New approaches to sulphur production, pipelines and melting
- Improving plant performance through effective maintenance planning and analysis

TECHNICAL PROGRAMME

Selected highlights

Feskekorka ('Fish Church') fish market, Gothenburg.

Claus waste heat boiler design

The waste heat boiler (WHB) is a critical piece of equipment in a sulphur recovery unit, but can often become the weak link in the SRU. Sulphur plant operators have recently been experiencing higher than normal WHB failure rates and increased attention is now being given to its design and operation. **Optimized Gas Treating** will examine various parameters that contribute to failures such as mass flux, amongst others, which are shown to have notable effects on WHB economics, reliability, safety and overall Claus unit performance.

Ceramic paper is an integral and necessary component of the tube sheet protection system of the waste heat boiler in sulphur recovery units. Appropriate permeability properties are needed for CFD simulation and the evaluation of tube sheet protection systems but are not currently available.

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Industrial Ceramics and nVent Thermal Management will present the results of a study conducted to determine gas permeability through ceramic fibre paper.

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Until now, the potential to utilise big data and artificial intelligence (AI) to manage sulphur transport pipelines has been largely untapped. TopSide Solutions and nVent Thermal Management will present an industry leading approach for a safe and reliable heated sulphur pipeline management program using customised software. Utilising pipeline operating data extracted from a fibre optic distributed temperature sensing (DTS) system on the pipeline, combined with other pipeline and electrical equipment instrumentation, decision-based outcomes become much more predictable by leveraging the enormous amount of available data.

New generation catalysts for enhanced sulphur recovery

Jacobs Comprimo[®] will present its new selective oxidation catalyst: EUROCLAUS[®] STRATA which improves the performance of the selective oxidation reactor, increasing the overall sulphur yield at this stage. Recent performance in various EUROCLAUS[®] installations and continuous improvements provide an interesting future perspective. What happens when a catalytic type process using the Claus reaction, hydrogenation and selective oxidation meet a recovery efficiency of >99.7%? What impact does this have on overall project economics as well as CO_2 emissions compared to amine based tail gas treatment units?

Euro Support and **Criterion** have joined forces to combine the Euro Support pure titania carrier with the Criterion CoMo impregnation technology to exploit the best of both worlds in creating the latest generation of low temperature high resistance tail gas catalysts. Euro Support will elaborate on the latest developments and provide a recap on the benefits of titaniabased tail gas hydrogenation catalysts. The more challenging the operational circumstances, the larger the benefits this new generation of catalysts can offer.

Challenging acid gas concentrations

Oil and gas fields in the Caspian region present a significant challenge due to their high H_2S concentrations. Given the current and projected sulphur supply surplus, alternative field development options, with-

PRE-CONFERENCE WORKSHOPS

Process infrastructure options for sulphur producers and consumers in the dynamic sulphur market

The global sulphur market is expected to continue to be oversupplied in the near future resulting in a marked impact for both producers and consumers along this vital supply chain. Oil and gas producers may be challenged to find acceptable prices for sulphur offtake and may consider pouring to block. Sulphur consumers however, may be able to reduce feedstock costs by diversifying between liquid and solid sulphur sourcing to obtain the best possible economics.

With the help of case studies including Matrix PDM's recent projects in China, Central Asia, North America and the Middle East to provide a global perspective, this workshop will include:

- Brief profile of world supply and demand centres for sulphur and sulphuric acid/ fertilizers
- Detailed discussion on molten sulphur handling and storage tank design and construction
- Process/equipment highlights for: sulphur recovery; degassing; forming and loading; blocking storage; remelters; sulphur unloading; melting and supply for sulphuric acid manufacturers
- Overview discussion of some leading technologies for SRUs and sulphur degassing, forming (50-10,000 t/d) and remelter (3,600 t/d in a single unit).

Raising project capital: Maximising export credit financing for your project

In today's fluctuating market conditions and capital constrained environment, economic viability and financing opportunities are of the utmost importance for businesses. Due to increasing competition within organizations for internal financing and in the world market for external project funding, traditional prerequisites do not necessarily lead to project realisation. Whether you are a major with a strong balance sheet or a junior developing a new site, securing project financing plays a crucial role in your overall success.

Throughout this year's workshop, SNC Lavalin's expert in export credit financing will lead delegates through the various possibilities to utilise export credit agencies (ECA) financing to build future projects.

SNC-Lavalin will be present an overview on the significant role that ECA financing has played on overall project development and achievement and will discuss key lessons learned and case studies demonstrating how project financing has been developed for challenging projects and situations.

Outotec's life-cycle philosophy to boost sulphuric acid plant profitability

Outotec is inviting conference attendees to participate in its workshop to share business experience, discuss industry challenges and consider potential solutions. This three-hour workshop aims to gather industry professionals to talk about the current and future market trends and common operational problems. The workshop will be split into two parts:

Part 1: Improving profitability – learning from the past:

- Impurities removal
- Heat recovery
- Digitalisation

Part 2: What's next for the industry – looking to the future:

- Plant availability
- Mega plants
- Oxygen enrichment
- Energy recovery

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out sulphur recovery must be considered. Reinjected acid gas after separation from the condensate and hydrocarbon gases may become an alternative to the standard approach in the development of these fields. **LUKOIL Engineering** will discuss the geological and technological challenges of such field development.

Sour gases containing low concentrations of H₂S can be particularly troublesome and costly to process in conventional SRUs. However, unique acid gas enrichment configurations can be utilised to achieve economical, robust operation in grassroots facilities, or can be employed in existing facilities that are required to meet new, lower SO₂ regulations. UniverSUL Consulting will evaluate operation and economics at leaner acid gas concentrations and different operating conditions, e.g. ultra-high tail gas ratio. Comparisons to other alternatives such as natural gas cofiring and oxygen enrichment will also be explored.

Water treatment in SRUs

As the world moves towards more sustainable processes with minimum impact on the environment, new ways of meeting stringent environmental regulations are being investigated and effluent streams are becoming of particular interest to plant designers and operators. Jacobs Comprimo® and Cool Separations will present a solution for the treatment of SRU water effluent streams which are difficult to handle by conventional water treatment solutions. In particular, water streams with a high sulphate content can be treated using an innovative process called eutectic freeze crystallisation (EFC), which makes use of freeze crystallisation up to the eutectic point. The benefits of this technology in combination with typical SRU effluent streams will be explored.

Claus tail gas treating

An important aspect of solvent selection in Claus tail gas treating is to maximise H_2S absorption while minimising CO_2 coabsorption. Selective treating permits full utilisation of the solvent for H_2S removal, thereby reducing circulation rate and increasing efficiency. **ExxonMobil** and **BASF SE** will present various design fundamentals for sulphur selective designs coupled with the latest solvent technology to achieve the lowest capex and opex.

Siirtec Nigi will provide information on the successful re-start-up of the High Claus Ratio[™] (HCR[™]) TGTU at Mullitah Oil & Gas

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(MOG) complex in Libya. The TGTU (originally commissioned in 2004) was shut down after a few years but has been put back into operation to comply with more stringent environmental regulations. Benefits to the SRU while operating the plant in HCRTM mode, enhancing plant availability and life will also be highlighted.

ASRL will describe experiments carried out to investigate the corrosivity of ammonium sulphur oxyanion salts that can be formed in the quench tower of a reducing tail gas unit under certain upset conditions. Ammonia is often added to the quench water for pH control, even if upstream ammonia has been destroyed. It was found that these salts are not corrosive and when further experiments were performed to investigate the influence of these salts on wet sulphur contact corrosion, it was observed that such salts either drastically reduce the typical rate of sulphur contact corrosion or completely shut it down. It was revealed that the ammonia generated from these salts was responsible for the corrosion inhibition.

Sulphur operations troubleshooting clinic

This interactive clinic, moderated by industry expert **Elmo Nasato**, will provide all participants with the opportunity to discuss, question and share experiences across a large range of operational scenarios. Themes explored will include:

sulphur recovery;

- tail gas treating;
- sour water stripping:
- contaminant destruction;
- maintenance and reliability:
- HSE strategies and best practice;
- sulphur handling.

New state-of-the-art sulphuric acid plant

Boliden's new sulphuric acid plant in Harjavalta, Finland, applies state-of-the-art technology to comply with stringent local emission restrictions and to produce premium quality sulphuric acid. Outotec will present details and characteristic data of the gas cleaning and sulphuric acid plant. The 2,240 t/d acid plant employs Outotec's proven 5-bed converter (3+2) with three integrated heat exchangers for optimum SO₂ conversion, operating with strong SO₂ feed gas of 14 vol-% and a guaranteed SO₂ emission of less than 100 ppm at the stack. Excess energy is recovered in the catalytic section to produce high pressure steam as byproduct, while waste energy recovered in the absorption section is used to generate hot water of

up to 95°C which is supplied to a nearby power station.

High speed replacement of a sulphuric acid drying tower

While the construction of drying towers in sulphuric acid plants can be time consuming, a presentation by **DuPont Clean Technologies** and **Glencore Nikkelverk** will demonstrate that it is possible to safely install a new tower in a shutdown window of seven days given good planning and collaboration between the plant operator and engineering/design suppliers. This presentation will trace the journey of the replacement tower development and installation with illustrations and graphics to explain the use of the 3D model, on-site fabrication and pre-assembly work.

Sulphuric acid production from pulp mill non condensable gases

Sulphur is an essential chemical element in Kraft pulp mills and actively participates in reactions with wood chips to produce pulp. Sulphur is present in black/white liquors and discharge waters and escapes the pulp mill processes as non-condensable gases (NCG). **Valmet** has developed wet gas sulphuric acid production technology from the incineration of NCG and will present the process and its environmental advantages. The first plant of this type started up in 2017 and has been operating continuously at Äänekoski, Finland.

Debottlenecking at Kansanshi copper smelter

The sulphuric acid plant at the Kansanshi copper smelter in Solwezi, Zambia had been operating for less than two years when a study was undertaken to expand the capacity of the plant. The acid plant was the bottleneck in the operation of the smelter. **Kansanshi Mining** will describe the process of identifying the bottlenecks and the work done to eliminate them.

Gas cleaning plant improvements at Atlantic Copper smelter

Atlantic Copper will present the operation and maintenance improvements that have been carried out in the wet gas cleaning plant at the Atlantic Copper metallurgical complex in Huelva, Spain. The improvements were aimed at increasing the cooling and cleaning capacity, ensuring the quality of the sulphuric acid and commercial gypsum and improving the rates of efficiency of operation and maintenance.

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Development of the mega sulphuric acid plant

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Phosphate fertilizer producers have an ever increasing demand for sulphuric acid to keep pace with capacity expansions. To meet this challenge, Chemetics has developed a novel sulphuric acid plant design that allows single train capacities in excess of 10,000 t/d. The design offers lower capex as well as enhanced energy recovery. Chemetics will describe the process features and provide key comparisons with conventional plant designs.

Safer heat recovery in sulphuric acid plants

SO₂ absorption heat recovery relies on absorbing the SO2 into highly concentrated acid to produce hot acid stream that is later cooled by a boiler. The failure of this boiler can be catastrophic. Clark Solutions will present a new heat recovery technology which addresses these issues, creating a scenario where SO_2 heat can be recovered as high pressure steam and, more importantly, virtually eliminating the corrosion/explosion risk associated with boiler corrosion and failure.

Aging assessments of stainless steel converters

Austenitic stainless steel SO_2 to SO_2 converters are common in sulphuric acid plants and most have been in operation for over 20 years. Norda Stelo will present results of thermomechanical analysis studies on the damage mechanisms that these converters are susceptible to and will discuss the importance of these measurements together with internal 3D scans to ensure long-term integrity and optimal capital spending, as well as enabling proactive maintenance planning and avoiding unplanned shutdowns.

Optimising tower design

Sulphuric acid plants with tower diameters that exceed those of the previous generation have experienced an increase in failures. With the increases in the circumference of these larger plants the coefficient of thermal expansion plays a larger role not only on the reliability of brick towers but also alloy towers. Koch Knight will discuss ways to both reduce the diameter of new towers and how to manage the thermal expansion in large diameter towers. Improved methods and materials of construction will be reviewed for both the absorption tower structure and the tower internals.

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Copper smelter supplying low CO₂ district heating

Aurubis Hamburg Copper Smelter has redesigned the intermediate absorption tower system of its sulphuric acid plant to supply industrial heat to a nearby new city district of Hamburg. Aurubis and Smart SCOPE will describe the whole project from conception to realisation. The project has earned the award "lighthouse project of Germany" for its public use of industrial heat with a very low CO_2 footprint.

Modular sulphuric acid plants

In January 2018 Outotec was awarded a contract to deliver three modular sulphuric acid plants for the Mutoshi copper-cobalt production complex in the Democratic Republic of Congo. In this presentation Outotec will focus on the delivery of the modular acid plants and the progress made to date on the project. The innovative plant concept and the benefits of modular prefabricated plant delivery, such as low investment, installation and operation costs will be discussed.



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Successful start-up of first ICOn degassing system

Controls Southeast Inc (CSI) recently started operation of the first commercial ICOn sulphur degassing system. ICOn is a new approach to sulphur degassing, utilising a packed-bed catalyst to remove H_2S from liquid sulphur to extremely low levels. Degassing is performed at low pressure and with a required residence time of less than five minutes. **Brandon Forbes** of CSI describes the ICOn system, its chemistry and advantages and reports on the successful results of the recent start-up of the first installation at a US Gulf Coast refinery.

he modified Claus process is universally used for large-scale sulphur recovery. Gas streams containing various sulphur compounds are processed into elemental sulphur produced in a liquid form. As a by-product of this process, the liquid sulphur contains high levels of H_2S . This H_2S will gradually leave the sulphur during storage, handling, and transportation. H_2S is highly toxic and explosive. Accumulation of H_2S in the head space of vessels and operator exposure are significant safety concerns for refineries.

ICOn degassing chemistry

The H_2S contained within the liquid sulphur exists in two chemical forms. Dissolved H_2S is simply H_2S molecules mixed in with the liquid sulphur molecules. This form of H_2S can be removed readily by agitation. When the dissolved H_2S is exposed to a vapour with a low H_2S concentration, it readily leaves the liquid in favour of the vapour. All existing degassing technologies use some form of sparging, spraying, or other agitation to remove the dissolved H_2S .

The second form of H_2S contained within the sulphur is chemically-bound H_2S ; this form is more challenging to remove. Above 159°C sulphur molecules form polymer chains. During the Claus process, these chains join with the available H_2S to form H_2S_x – which is analogous to a sulphur polymer chain capped with a hydrogen sulphide molecule. These chains are unstable and will slowly break down into elemental sulphur and H₂S. But this process is very slow at atmospheric pressure and is equilibrium-limited by the surrounding dissolved H₂S. The ICOn degassing approach utilises a catalyst to accelerate the decomposition of the chemically-bound H₂S while simultaneously using a sparge



gas to remove the dissolved H_2S (Fig. 1). This '1-2-punch' approach fully degasses the sulphur with less than five minutes residence time.

The catalyst is in the form of a packed bed and is not consumed. The catalyst is expected to last at least from unit turnaround to turn-around. Barring a major upset condition in the SRU, it is conceivable that the catalyst will last indefinitely.

The role of temperature

Sulphur freezes at 120°C. Liquid sulphur above this temperature takes on a molecular form analogous to a stop sign comprised of eight sulphur atoms. But at temperatures above 159°C, this stop sign opens up and the sulphur molecules form polymer chains. This 'open chain' form of sulphur readily bonds to H_2S – effectively preventing degassing. Thus, degassing can only be performed in the 120°C to 159°C temperature window.

Unfortunately, the sulphur exiting the Claus unit condensers is often hotter than 159°C. Some form of sulphur cooling is typically required. If the ICOn system is installed downstream of sulphur storage (typically a collection vessel or pit), it is likely that the sulphur will cool down adequately and can be sent directly to degassing. But if the sulphur is sent directly from the Claus condensers to degassing, a sulphur cooler will be required.

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The heart of the ICOn degassing system is the contactor (or reactor). The contactor contains the packed-bed catalyst and the vapour sparge system. Liquid sulphur is sparged in the presence of the catalyst to achieve the '1-2-punch' degassing. The contactor has four major nozzles: liquid in/out, and vapour in/out. There are many options for the arrangement of equipment (Fig. 2) that feeds and return these process streams. A few are described here.

Pumped liquid: This configuration is traditional for retrofit degassing technologies. The sulphur is pumped out of the Claus unit pit (or collection vessel) to the degassing contactor. The sulphur in the pit remains un-degassed; the pit vapour space remains a collection point for accumulation of dangerous H_2S gases.

A variation of this arrangement also exists in which the sulphur is collected in a vessel whose vapour space is operatively connected to one of the Claus condensers. This allows the collection vessel to be sealed and significantly reduces the concerns associated with H_2S accumulation.

Pumpless liquid: This is a novel approach pioneered by CSI that places a sulphur cooler in the Claus run-down lines. The cooler is specially designed to create minimal pressure drop. The pressure and elevation differential between the Claus condensers and the ICOn contactor is adequate to move the sulphur through the cooler. Sulphur flows directly from the condensers, through the cooler, into the contactor. No pump or intermediate storage is required.

In this configuration, only sulphur from the first two or three of the four Claus condensers requires degassing. Sulphur from the remaining condenser(s) has a sufficiently low H_2S content and flow rate that, when combined with the sulphur from the ICOn contactor, the net H_2S content is still below the 10 ppm industry standard.

Process sparge: On the vapour side, tail gas from the Claus unit can be used as the sparge gas. The H_2S content of tail gas is low enough that its impact on the degassing chemistry is negligible. A slip-stream is taken from the tail gas line and returned to the Claus unit, typically just upstream of the last re-heater. An ejector is required to pull the process gas through the contactor and raise the pressure adequately to return it to the Claus unit.

Air sparge: An alternative arrangement is to use atmospheric air for the sparge gas. The air source can be plant air, or a slipstream from the Claus furnace blower. The air leaving the contactor is sent directly to the Claus furnace and supplements the normal furnace air supply. With this configuration, the contactor operating pressure must be higher than that of the Claus furnace. Thus, a pump is required on the liquid side to get sulphur into the contactor. An advantage of this configuration is that the oxygen in the air acts as a supplement to the catalyst, shortening the required residence time. Thus, a smaller contactor can be used. Other sparge gases can be used including steam and nitrogen. Various vapour return points can be considered depending on the choice of sparge gas.

ICOn system advantages

Degassing the sulphur makes it safer to handle and transport, reduces emissions from sulphur storage, and simplifies the downstream equipment. These benefits are provided by any sulphur degassing system. ICOn provides several additional and unique benefits:

- The sulphur can be degassed immediately as it exits the Claus condensers (depending on the chosen equipment arrangement). Thus, no storage of undegassed sulphur is required at any location in the refinery. This reduces the hazards associated with H₂S accumulation in the vapour space and reduces plant emissions.
- The waste stream from the contactor is returned directly to the Claus unit where it is processed with the rest of the sour gas. Thus, there is no increase in emissions and no requirement for additional scrubbing equipment.
- The sparge gas being sent to the Claus unit has only a very small impact on overall capacity and conversion in the SRU and TGU. With some equipment configurations, such as using air sparge, the impact is immeasurable.
- The amount of rotating equipment is minimised (the extent of which is dependent on the chosen equipment arrangement), thus reducing maintenance requirements and potential down-time.
- The exact arrangement of equipment includes many options which can be tailored to the specific needs of each individual refinery, thus reducing cost by providing a system comprised of only what's needed.
- The catalyst is a solid that is not consumed, thus maximising reliability and minimising contactor maintenance.

First commercial ICOn system

The first ICOn sulphur degassing system recently started up at a US Gulf Coast refinery, which is currently processing 300 t/d of liquid sulphur, dropping the H₂S level in the sulphur from 350 ppm to 2 ppm. Refinery management is now pursing the installation of similar ICOn degassing units for their remaining sulphur recovery units.

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Energy efficiency vs sulphur and carbon management

Claus sulphur recovery provides significant energy benefits which should be leveraged, but striving for ever higher sulphur recovery efficiencies can erode these benefits. **A. Slavens** and **S. Khan** of UniverSUL Consulting discuss energy production and consumption in the sulphur plant and compare different sulphur recovery technologies with regard to energy efficiency, SO_2 emissions and carbon footprint.

he sulphur recovery facility within a refinery or gas plant is required to meet SO_2 emissions regulations and is often viewed as a cost of production. However, waste heat from the exothermic Claus reaction is recovered as HP and LP steam, which usually makes the sulphur plant a net energy exporter, supplying needed steam and/or power to the processing complex.

The current industry sulphur recovery efficiency (SRE) benchmark is around 99.9%. However, this figure is on the rise with a greater number of facilities designing for higher sulphur removal rates, as evidenced by the World Bank Standard (WBS), which currently sits at 150 mg/ Nm³ (equivalent to approximately 99.98% recovery efficiency). While additional SO₂ emissions reduction is beneficial for reduc-

ing harmful environmental impacts, there is cause to question whether striving for ever higher recovery efficiencies is actually leading to diminishing returns, in terms of increased energy consumption and associated carbon emissions.

Energy production and consumption in the sulphur plant

The Modified Claus process is shown in Fig. 1. In this well-known process, one third of the H_2S in the acid gas is burned to form SO_2 , which then reacts with remaining H_2S to form elemental sulphur, via the exothermic Claus reaction. Key utilities produced/ consumed in the process are steam (HP and LP), fuel gas and electric power. Sulphur recovery efficiencies of 95-97% are achievable with a standard Claus sulphur

recovery unit (SRU). Typically, some form of tail gas treating is required downstream of the SRU to achieve sulphur recovery efficiencies of 99% and above, the energy requirements of which can be substantial.

Steam

As shown in Fig. 1, heat released in the process is recovered in the SRU waste heat boiler (WHB) as HP or MP steam, and in the sulphur condensers as LP or LLP steam. In addition to heat released by the Claus reaction, the incinerator produces heat via combustion of fuel gas to achieve temperatures hot enough to ensure complete oxidation of H_2S in the tail gas stream. Incinerators are often equipped with waste heat boilers and/or HP steam superheaters to recover some of this heat and maximise process efficiency.



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SRU ENERGY EFFICIENCY

Steam consumers in the process include feed gas preheaters and process gas reheaters, all of which typically utilise HP saturated steam. The SRU and incinerator air blowers may also consume HP steam if steam turbine drives are employed. The only continuous LP steam consumer is the reboiler in an aminebased tail gas treatment unit (TGTU).

Overall, the sulphur recovery facility is a net HP steam exporter. It is typically also an LP steam exporter; however, this may not be the case for amine-based TGTUs with extremely high recovery efficiency requirements, which can consume all (or more) of the LP steam produced in the SRU. This is usually not the case unless SRE significantly exceeds 99.9%.

Fuel gas

The incinerator is a continuous fuel gas consumer. Fuel gas is burned with excess air and the combustion effluent is mixed with SRU tail gas to achieve a minimum temperature of 650°C for nearly complete oxidation of H₂S to SO₂. Sometimes higher temperatures are required to achieve lower limits on CO and/or total reduced sulphur (TRS), up to a maximum of around 815°C.

In some facilities which process lean acid gas, continuous fuel gas co-firing may be employed in the SRU burner to achieve temperatures high enough for BTEX destruction. Other methods for increasing furnace temperature such as acid gas enrichment (AGE) or oxygen enrichment are preferred, as they reduce the risk of soot deposition and/or fire in the downstream catalyst beds, as well as minimising the process gas flow through the facility, thereby minimising the size of equipment and piping. Nevertheless, fuel co-firing is not an uncommon practice for increasing furnace temperature.

In older facilities, fuel gas is sometimes consumed in SRU fired reheaters; however, most modern SRUs utilise indirect HP steam reheaters to avoid the concerns mentioned above for fuel co-firing in the SRU burner. Most modern amine-based TGTUs employ preheating with HP saturated steam upstream of the hydrogenation reactor. However, for facilities that are not equipped with a hydrogen source, reducing gas generators (RGGs) are often installed. In an RGG, fuel is combusted sub-stoichiometrically to produce reducing gas; the exhaust gas is then mixed with the SRU tail gas to achieve sufficient temperature for the hydrogenation and hydrolysis reactions to occur in the downstream reactor. RGGs result in increased

Table 1: Feedstock for 1,000 t/d benchmark plant

Component	mol-%	kmol/h
H ₂ S	60	1,300
C0 ₂	30	650
Hydrocarbon (as C1)	1	22
H ₂ O	9	195
Total	100	2,167
Temperature, °C		54
Pressure, barg	C	0.69

Source: UniverSUL Consulting

energy consumption (vs TGTU steam preheaters) due to fuel consumption in the burner and also result in increased process gas flow through all equipment downstream of the RGG.

Overall, the sulphur recovery facility is a net fuel gas importer. All SRUs require continuous fuel firing in the incinerator; however, facilities which employ continuous fuel firing in the SRU burner, reheaters and/ or TGTU RGG may require significantly more fuel consumption than units which do not.

Electric power

The Claus and incinerator blowers are the primary electric power consumers in a sulphur recovery facility, when these machines are equipped with motor drivers. Other power consumers include air-cooled heat exchangers and pumps. In hot climates and/or when extremely high sulphur recovery efficiency is required, refrigeration may be required for solvent and quench water cooling in the TGTU.

Overall, the sulphur recovery facility is a net power importer. Facilities which employ amine-based tail gas treating may utilise significantly more power than those which do not, due to additional air-cooled exchangers, pumps and possible refrigeration utilised in those facilities.

Comparison of sulphur recovery technologies

The overall impact of the various utility producers and consumers described is that the sulphur recovery facility is typically a net energy exporter, although the quantity of energy exported can vary greatly depending on the type of tail gas treating technology employed. In some cases, the facility may actually need to import energy when very high sulphur recovery efficiency is required,

negating the energy benefits of the Claus process.

To illustrate this, a hypothetical 1,000 t/d sulphur recovery train, which will be referred to as the benchmark plant, is considered over a range of sulphur recovery efficiencies. Considering that most refineries produce rich acid gas ($H_2S > 85$ mol-%) and most gas plants produce relatively lean acid gas (40-50 mol-% H_2S), a median concentration of 60 mol-% is assumed. Feed gas flow and composition for the hypothetical plant are provided in Table 1.

To compare relative energy balances for varying recovery efficiencies, simulations are generated for a range of tail gas treating technologies and plant configurations. The following SRE cases are explored.

- A: 97% SRE 97% recovery is based on a conventional 3-stage Claus unit.
- B: 99.0% SRE 99.0% recovery is based on a sub-dewpoint process (2-stage Claus + 2 sub-dewpoint reactors), although it should be noted that a direct oxidation process would achieve similar SRE and energy balance.
- C: 99.3% SRE 99.3% recovery is based on a 2-stage Claus unit + TGTU (MDEA). This SRE is just beyond the upper limit of an achievable guarantee value for subdewpoint and direct oxidation processes; therefore, it is investigated as the entry point for an amine-based TGTU.
- D: 99.9% SRE 99.9% recovery is based on a 2-stage Claus unit + TGTU (MDEA).
- E: 150 mg SO₂/Nm³ (MDEA) The World Bank Standard case (99.98% SRE) is first investigated based on a 2-stage Claus unit + TGTU with generic solvent (MDEA).
- F: 150 mg SO₂/Nm³ (Proprietary Solvent) The World Bank Standard case (99.98% SRE) is investigated utilising a more selective solvent in the TGTU and corresponding positive energy impact; thus, this case is based on a 2-stage Claus unit + TGTU with proprietary solvent.

Process flow diagrams for the six cases are provided in Figs 2-5.

A standard set of design parameters is employed for all cases to allow relative comparison on a consistent basis. Key design features are aimed at optimising energy efficiency, as follows:

Sulphur recovery unit

- air-only operation, without fuel co-firing;
- motor-driven Claus air blowers;
- HP saturated steam (40 barg) produced • in SRU waste heat boiler (WHB);

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Table 2: 1,000 t/d benchmark plant process parameters

Case	Α	В	С	D	E	F
SRE	97%	99.0%	99.3%	99.9 %	99.98%	99.98%
Stack gas SO ₂ (kmol/h)	38.95	12.97	9.04	1.24	0.28	0.28
Stack gas CO ₂ (kmol/h)	870.6	885.7	845.4	850.6	851.2	851.2
Total stack gas flow (kmol/h)	8,012	8,134	6,176	6,188	6,189	6,189
Amine circulation (m ³ /h)	-	-	176	264	1,026	513

Source: UniverSUL Consulting

- 2 Claus beds (3 for 97% SRE case) with promoted activated alumina catalyst;
- 2 additional sub-dewpoint beds for 99.0% SRE case;
- LP steam (3.5 barg) produced in first and second sulphur condensers;
- LLP steam (1.0 barg) produced in third and fourth sulphur condensers;
- HP saturated steam (40 barg) consumed in SRU preheaters and reheaters.

Amine-based tail gas treatment unit

- HP saturated steam (40 barg) consumed in preheater;
- low temperature hydrogenation catalyst (232°C inlet temperature);
- LP steam (3.5 barg) produced in TGTU waste heat exchanger (WHE);
- Lean solvent temperature of 50°C for all except Cases E and F, which was reduced to 40°C to achieve ultra-high SRE (air cooling to 50°C with CW trim cooling);
- LP steam (3.5 barg) consumed in regenerator reboiler;

 solvent circulation rate for Case F assumed as 50% of Case E to approximate proprietary solvent.

Incinerator

- Operated at 815°C (upper limit, required for achieving <5 mg/Nm³ TRS);
- 2% excess O₂ in stack gas;
- fuel fired (LHV of 8,953 kcal/Nm³);
- motor-driven incinerator air blowers;
- HP saturated steam (40 barg) produced in incinerator WHB;

• no sulphur pit ejector routed to incinerator. The SO_2 and CO_2 content of the incinerator stack gas for the range of SRE cases is summarised in Table 2. For the aminebased TGTU cases, amine circulation rate is also provided for information.

Energy balance of benchmark plant

Thermal energy production/consumption figures for each of the benchmark plant case studies are summarised by key utilities in Table 3 and by unit operation in Fig. 6. For electric power, the equivalent thermal energy consumption is calculated based on electricity generation using a steam turbine with an overall thermal to electric energy conversion efficiency of about 43%.

It is observed that as SRE increases, energy export decreases, and the facility reverts from net energy export to import at ultra-high recovery efficiency (Case E). The balances clearly illustrate that the SRU is always a net energy exporter whose energy production remains fairly constant, even for Case B, which employs a non-amine-based tail gas treating technology to achieve higher SRE.

It is the amine-based TGTU that is responsible for increasing energy consumption as SRE increases, primarily due to LP steam consumption for MDEA solvent regeneration in the TGTU. When a highly selective proprietary solvent is employed (Case F), LP steam consumption and solvent cooling requirements are reduced substantially, making the facility closer to energy-neutral, but the overall impact of the TGTU on sulphur recovery energy production is still significant.

The world average sulphur recovery efficiency for new plants is around 99.9% (Case D), which results in a 44% energy penalty on the standalone Claus plant. Despite its detrimental impact on the overall SRU/TGTU energy balance, the aminebased tail gas treating process is currently the only conventional technology available

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for achieving guaranteed SRE in excess of about 99.3%.

Energy KPIs for benchmark plant

The net energy balance figures provided in Table 3 are converted to "thumb rule" targets that can be used to assess a sulphur recovery facility's energy performance, as provided in Table 4 and Fig. 7. These key performance indicators (KPIs) can be utilised by operators to evaluate whether their facilities are operating in accordance with best energy efficiency standards.

It is important to keep in mind specific feed conditions and plant design configuration when applying this information. A different configuration, feedstock and/ or operating philosophy can lead to significant variations in KPIs. For example, a plant which is equipped with an incinerator WHB can generate up to 40% more HP

Table 3: Benchmark plant energy balance by utility (MW)							
Case	A	в	с	D	Е	F	
SRE	97%	99.0%	99.3%	99.9%	99.98%	99.98%	
Utility	· · ·						
HP steam	+77.4	+78.8	+66.8	+67.0	+67.0	+67.0	
LP/LLP steam	+27.2	+27.9	+16.9	+10.6	-57.4	-14.0	
Fuel gas	-43.8	-47.1	-38.3	-39.4	-39.6	-39.6	
Electric power	-4.1	-4.1	-6.1	-6.5	-9.9	-7.6	
Cooling water	-	-	-	-	-27.3	-13.6	
Net energy import/export	+56.6	+54.4	+39.3	+31.8	-67.1	-7.9	
Comparison to Case A	-	- 2 %	- 31 %	-44%	-219%	-114%	

Source: UniverSUL Consulting

Table 4: Energy performance KPIs for benchmark plant

Case	Α	в	с	D	E	F
SRE	97%	99.0%	99.3%	99.9%	99.98%	99.98%
kWh/tonne 'S' produced	+1,400	+1,434	+949	+764	-1,612	-189
kWh/Nm ³ H ₂ S in acid gas feed	+1.94	+1.90	+1.35	+1.09	-2.31	-0.27
Source: UniverSUL Consulting						

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steam than one that is not. Some other examples that can lead to widely varying KPIs include fuel gas co-firing in the SRU, the use of an RGG in the TGTU, installation of low-temperature catalyst in the TGTU and TGTU solvent chilling requirements, to name a few.

For the most part, the energy efficiency of the benchmark plant design has been optimised across all Cases A-F, with the exception of incinerator operating temperature, which could be reduced by around 165°C, depending on the emission regulations. However, since an incinerator WHB is employed in the benchmark plant design, additional waste heat is recovered at the higher temperature and the overall impact on energy efficiency is negligible.

There are some other minor opportunities for improving energy efficiency that are not included in the benchmark plant design, such as BFW preheat in the final condenser and sulphur cooler to maximise HP steam production (rather than generating LLP steam), but the overall impact on energy export/import for these items is not expected to significantly impact the results of this study.

CO₂ emissions from benchmark plant

When considering the carbon footprint of a sulphur recovery facility, it is important to look beyond the obvious CO_2 content of the stack gas. It is also essential to take into account the equivalent CO_2 emissions associated with all of the major energy producers and consumers in the facility.

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The energy values from Table 3 are used to calculate the net equivalent CO_2 emissions for each of the cases studied. Fig. 8 summarises the SO_2 and CO_2 emissions from the benchmark plant. Because a sulphur recovery facility is normally an energy exporter, net CO_2 equivalent is lower than the actual CO_2 value in the flue gas for all except Case E, in which the TGTU utilises generic MDEA rather than a highly selective solvent.

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 SO_2 emissions decrease substantially from Case A to D, while net CO_2 emissions only increase by about 20%. However, in increasing sulphur recovery from 99.9% in Case D to 99.98% in Case E, there is only a very minor decrease in SO_2 emissions with a significant corresponding CO_2 increase of 50%. When a highly selective proprietary solvent is employed (Case F), the corresponding CO_2 emissions increase is lower but still significant at about 20%.

The dramatic increase in CO_2 emissions for only a marginal decrease in SO_2 emissions can be more clearly illustrated with the correlations provided below.

When increasing SRE from 99.0% to 99.9%:

- every tonne of SO₂ reduction results in five tonnes of CO₂ emissions, or;
- every tonne of S reduction results in ten tonnes of CO₂ emissions.

When increasing SRE from 99.9% to 150 $\rm mg/Nm^3$ (MDEA):

- every tonne of SO₂ reduction results in 240 tonnes of CO₂ emissions, or
- every tonne of S reduction results in 480 tonnes of CO₂ emissions.

The impact of increasing SRE to ultra-high values, in excess of 99.9%, clearly has a negative impact on CO_2 emissions. While SO_2 emissions have an immediate impact near the pollution source, it is believed that CO_2 is more likely to have long-term effects on the global environment, which begs the question as to whether the minor SO_2 reduction benefit is justified.

Impact of sulphur recovery facilities on global and local SO₂ and CO₂ emissions

Total global anthropogenic SO_2 and CO_2 emissions in recent years were in the range of 120 million t/a and 36,000 million t/a respectively¹, while world elemental sulphur production in 2016 was approximately 63.4 million tonnes². Scaling up from the 1,000 t/d benchmark plant to a global production of 63.4 million t/a gives SO_2 and equivalent CO_2 emissions as a percentage of world totals as shown in Fig. 9.

Assuming a world average sulphur recovery efficiency in the range of 99.5-99.9%, Fig. 9 illustrates that sulphur recovery facilities contribute somewhere around 0.5% of global SO₂ emissions and about 0.12% of global CO₂ emissions. Increasing recovery efficiency from 99.3% to 99.9% with an amine-based TGTU (Case C to Case D) only very slightly increases CO₂ emissions. For this reason, if amine-based tail gas treating is employed, it is certainly worthwhile to design for at least 99.9% SRE, from both an energy efficiency and CO₂ footprint perspective.

ISSUE 378 SULPHUR SEPTEMBER-OCTOBER 2018 Similar to what was observed in Fig. 8, increasing SRE to greater than 99.9% (Cases E and F) achieves little benefit with respect to SO_2 emissions but has a significant detrimental impact on CO_2 emissions.

SRUs located in some of the world's most significant sulphur-producing regions contribute a greater percentage to regional SO_2 and CO_2 emissions than the world average, due to a high level of industrial activity in those locations. For example, contribution from Middle Eastern SRUs to local SO₂ emissions is in the range of 2-3%, an order of magnitude greater than the contribution of all SRUs to the world average. Middle Eastern SRUs contribute greater than 0.2% of local CO₂ emissions, approximately double the world average. China is an exception, with sulphur recovery facilities contributing to less than 0.3% of local SO_2 emissions and only 0.05% of local CO2 emissions due to the large quantities of these pollutants emitted from coal-fired power plants. When compared to the world average, North American SRUs have a less significant impact on local SO2 emissions (less than 0.2%) due to relatively high recovery efficiency requirements in the region.

Therefore, SO₂ emissions specifications in excess of 99.9% may be considered in the case of very large sulphur recovery facilities that would have substantial SO₂ point source emission rates (t/d basis) and/or facilities that are located in environmentally sensitive regions. Conversely, there may be opportunities to relax SRE requirements below 99.9% for smaller SRUs (<50 t/d), which have only a

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Table 5: Energy balance by processing unit for 99.9% SRE cases (kW)

Case	D	G	н
SRE	99.9%	99.9%	99.9%
SRU	+61,992	+37,870	+37,195
TGTU	-20,712	0	0
Incinerator	-9,460	0	C
WSA	0	+72,907	+70,279
Net	+31,819	+110,777	+107,474

Source: UniverSUL Consulting

minor point-source emission impact, even at lower SRE. This philosophy has already been adopted in some parts of the world.

Alternative tail gas treating technologies

The authors are observing an increasing trend of operators wishing to make the best use of the sulphur recovery unit's energy benefits. As a result, SRU/TGTU energy efficiency is being increasingly evaluated and scrutinised, particularly for those sour facilities with relatively large sulphur recovery requirements. Since conventional tail gas treating technologies can significantly erode energy benefits provided by Claus SRUs, alternative technologies are being considered. For example, in the wet gas sulphuric

acid (WSA) process, residual sulphur in the Claus tail gas is recovered as concentrated sulphuric acid. The oxidation process is exothermic and no solvent regeneration is required; hence, energy performance should be improved, versus the amine-based tail gas treating process. A process flow diagram for a typical WSA unit, indicating the top utility producers/ consumers, is provided in Fig. 10.

Two cases for Claus tail gas treatment via the WSA process are considered for energy performance comparison, using the same acid gas feed flow and composition as the benchmark plant, and an SRE of 99.9%. Case G features a 1-bed Claus plant followed by a WSA unit, while Case H features a 2-bed Claus plant followed by a WSA unit. Both cases require acid gas bypass around the SRU to avoid

Fig. 11: Net energy import/export



continuous fuel gas consumption in the tail gas burner and to avoid the production of dilute acid.

As shown in Table 5, compared to Case D of the Benchmark Plant, which uses an amine-based TGTU to achieve a similar SRE, the WSA cases can reach the same extent of sulphur recovery with more than three times the net energy export. Net energy export from the SRU is reduced in the WSA cases due to acid gas bypass; however, the tail gas treating section of the process is converted from an energy consumer to an energy producer when WSA is employed. The primary reasons for this are the avoidance of continuous fuel consumption in the incinerator, combined with improved efficiency in the tail gas combustion heat recovery system, as well as elimination of the requirement for LP

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steam for solvent regeneration. The WSA cases consume more electric power due to the additional air and process gas blowers, and also have some additional energy requirements for acid cooling; however, the net result is that the WSA cases improve the energy efficiency of the facility.

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A comparison of the net energy import/ export across all of the sulphur recovery technologies and SREs considered is provided in Fig. 11. In comparing Case C to Cases A and B, it is apparent that the energyconsuming, amine-based tail gas treating unit erodes the energy benefits of the SRU. As SRE increases (Cases D and E), energy requirements for the TGTU also increase, resulting in an even greater negative impact on the overall energy balance. The use of a proprietary solvent in the TGTU (Case F) reduces TGTU energy consumption but it is still a net energy consumer. Because of the fact that the WSA tail gas treating facility is a significant energy producer, the energy balance of the overall sulphur recovery facility is enhanced to nearly double the SRU energy export (Cases G and H).

It is important to note that in both WSA cases, the sulphur recovery facility will convert slightly less than half of the H_2S in the acid gas feed to sulphuric acid, rather than elemental sulphur. This will have to be accommodated when considering product storage, transportation and marketing requirements, which will obviously have an impact on capital and operating costs; however, the energy impact will be minimal and will not affect the outcome of this evaluation. Depending on the location of the facility, marketing limitations may preclude a sulphuric-acid-producing option from being considered.

Conclusions

Sulphur recovery facilities provide significant energy benefits and should be leveraged to their fullest potential via astute design and optimised operation, deliberately focused on energy conservation and reduced equivalent CO_2 emissions. For extremely sour gas plants or refineries processing sour crude feedstock, the sulphur plant may be one of the areas of greatest interest for improving energy efficiency and strengthening the economics of production. Conventional tail gas treating technologies can significantly erode energy benefits provided by Claus SRUs and therefore should be

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designed and operated to achieve lowest acceptable SRE (highest acceptable stack gas SO₂ content). Given the huge energy requirements and carbon footprint of amine-based TGTUs, there may even be a case to lobby for relaxed SO₂ emissions regulations for future facilities³. However, this is a lofty goal, and should it prove unattainable, alternate technologies may be considered. In the case of WSA, the production of an additional sulphuric acid product would require careful consideration in terms of storage, transportation and marketing.

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At the forefront of sulphur granulation

IPCO's recent technological advancements in drum granulation have resulted in the SG20 sulphur granulation system. Developed for mediumcapacity units, SG20 technology benefits include reduced maintenance, longer run times and reduced sulphur particulate emissions.

PCO is a world-leading supplier of sulphur processing equipment including the Rotoform[®] technology for low-tomedium capacity granulation operations and fully automated sulphur drum granulators for high capacity sulphur granulation.

Previously operating as Sandvik Process Systems, the company is now an independent company within the Wallenberg group and has 600 employees, more than 35 sales and service offices and annual sales exceeding €200 million.

The SG20 sulphur granulation system (Fig. 1) is IPCO's newest addition to its wide range of sulphur processing technologies. Demand for a medium-capacity drum granulator, combined with the technological breakthroughs of the larger IPCO SG30 (formerly known as the Sandvik/Brimrock RS-1500), have led to the development of the SG20. The SG20 is a scaled-down version of the high capacity SG30.

The benefits of making strong, dry sulphur product are well known in the industry. A strong product (i.e. low-friability) is less prone to sulphur dust generation. Minimising dust generation has positive consequences for health, safety, and environmental aspects of materials handling operations. A dry sulphur product (i.e. less than 0.5% moisture) avoids the major issues associated with sulphuric acid, which is created by bacteria over time when you mix sulphur, water, and air. Dry product also minimises the energy consumption associated with melting the sulphur, which is the fate of most sulphur via consumption by the chemical industry.

All drum granulation processes follow the same basic principle of enlarging small particles of sulphur (called seeds) into full-size granules. The main differences in drum granulation processes come from the following:

- single pass vs multi-pass;
- seed generation;
- sulphur particulate emissions scrubbing.

These differences impact important parameters such as electrical power consumption, steam consumption, emissions, and installation footprint.

SG20 Process

The SG20, like its predecessor the SG30, is designed to solidify sulphur granules in the most efficient and environmentally-friendly way possible. For drum granulation technologies, this means simplifying the process down to only the essential components while properly managing sulphur particulate emissions to keep them as low as possible.

The liquid sulphur is supplied to the granulation system via a heated piping

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Fig. 1: The SG20 sulphur granulation system, image by Benjamin Herwig, IPCO Germany GmbH, Fellbach.

> system. The sulphur flow is then split into two streams, one stream to the seed generation system and the other stream to the rotating drum.

> Small solid sulphur seeds (the nuclei of the granules) are generated by freezing sprays of liquid sulphur in a water tank at controlled pressures to form the desired size range (Fig. 2). These particles settle to the bottom of the water tank are then gently transported into the granulation drum with a screw conveyor (Fig. 3). The drum has flights attached to its inner surface that pick up the seeds and drop them to create curtains of particles inside the drum.

> The seeds are progressively enlarged to the final product size by coating them multiple times with sprays of liquid sulphur inside the drum. The temperature inside the drum is moderated by the evaporation of water, which is provided by water spray nozzles.

> By the time the sulphur particles reach the end of the drum, they are within the desired product size range and are discharged onto a collecting conveyor (Fig. 4).

> A fan is used to draw a stream of air through the drum to sweep out the water vapour. Any sulphur particles entrained in the airflow are scrubbed out of this exhaust stream using a wet scrubber before the process air stream is released to the atmosphere.

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The sulphur particulates captured in the wet scrubber are pumped to the same water tank that is used to generate the seed particles. Here, the recovered particles settle out and extracted along with the seeds to be consumed in the process.

SG20 process comparison

Single pass granulation technology – IPCO sulphur drum granulators utilise a oncethrough process in terms of making small sulphur seeds, enlarging the seeds by spraying them with liquid sulphur multiple times in the drum, and then discharging the full-size granules from the system. Historical multipass systems use screens and recycle conveyors to move undersized product back to the front of the drum. Single pass has the advantage of requiring less equipment to achieve the same end results. This means a smaller footprint, faster installation, a simpler process to control, lower installation costs, and less maintenance.

External seed generation – IPCO drum granulators are the only technologies that create seeds external to the drum. All other processes create seeds internally

by using intersecting sulphur and water sprays inside the drum. These intersecting sprays need to be carefully aligned and controlled to avoid impacting product quality. The intersecting sprays also increase the required spray pressure and use smaller nozzles that are more prone to plugging. Additionally, these internal seed generation systems are limited to only processing cooler sulphur.

External seed generation provides the following benefits:

- creates a much steadier process that can handle upstream fluctuations (easier to control);
- allows for lower sulphur spray pressures and larger nozzles (lower pumping pressure and nozzles that are less prone to plugging);
- allows for recycling of the sulphur particulate captured in the emissions treatment system (lower steam consumption);
- lower sulphur spray pressures allow for lower sulphur particulate emissions (lower environmental impact);
- can process any liquid sulphur temperature up to 160°C (more flexibility).

Wet scrubbing for sulphur particulate capture - all drum granulation technologies pass air through the drum to remove evaporated water. Inevitably, some sulphur particulate is captured in this airstream which needs to be removed before the air is released to the atmosphere. There are multiple ways to remove sulphur particulate from the emissions of a drum granulator. Wet scrubbing has proven itself to be the most effective method. Some other processes use heated cyclones. Heated cyclones have the benefit of reducing the amount of rotating equipment. Unfortunately, this single benefit is outweighed by the drawbacks of higher sulphur emissions and much higher steam consumption. The SG20 employs a wet scrubbing technology.

Combine wet scrubbing with the lowest sulphur spray pressures of any drum granulation technology and this results in the lowest sulphur particulate emissions in the industry.

Recycled sulphur fines – the purpose of a drum granulator is to freeze sulphur. Simplifying drum granulation means developing a process where it is not necessary to melt sulphur as part of regular operation.

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0 10 20 30 40 50 ambient temperature, °C -- 130°C sulphur, 33% RH -- 130°C sulphur, 66% RH -- 130°C sulphur, 100% RH -- 140°C sulphur, 33% RH -- 140°C sulphur, 66% RH -- 140°C sulphur, 100% RH

Older technologies with wet scrubbers

(Fig. 5) capture the sulphur particulate

from the drum effluent and melt it. This

requires a significant amount of steam to

first boil off the high levels of water mixed

in with this sulphur and then melt the

sulphur. Older technologies with heated

cyclones (Fig. 6) capture the sulphur par-

ticulate and also melt it. At the same time,

they heat up the entire airstream from the

drum. This consumes more steam than

(Fig. 7) take a much more energy efficient

approach and recycle this sulphur particu-

late into the front end of the process to

use it as seed for creating granules. The

SG20 does not melt any sulphur as part of

Level drum for minimal maintenance -

granulation drums are heavy pieces of

rotating equipment. They are required to

rotate for two reasons: 1) to form curtains

of falling sulphur granules in the drum,

Further IPCO drum granulator

the forming process.

technology benefits

IPCO's granulation technologies

any other drum granulation technology.

and 2) to advance the granules from the inlet to the outlet.

Most technologies will place the drum on an angle to use gravity to advance the granules. This creates high levels of stress on the drum support system and creates alignment issues that require ongoing maintenance. As with all IPCO drum granulators, the SG20 sulphur granulation system has a completely level drum with advancing flights. This means no unnecessary wear and tear on the drum support system, and no ongoing maintenance to keep the unit aligned.

Minimal time for operators in the drum – traditional drum granulators require frequent cleanouts, often requiring daily shutdowns. This is a major burden on both the operators and the overall availability of the forming operation. IPCO granulators ensure that operators only need to go into the drum once per week, or less, providing the longest continuous run times and highest availability of any drum granulation technology on the market. The SG20 has been designed to minimise/eliminate sulphur build-up inside the granulation system.

SG20 performance

As with all drum granulation technologies that are solidifying sulphur, the production capacity is impacted by a wide range of factors. The most important factors are:

- temperature of liquid sulphur at the inlet
- temperature of ambient air
- relative humidity (RH) of ambient air

Fig. 8 displays the SG20 forming capacity for a range of inlet sulphur temperatures and ambient conditions.

Conclusion

IPCO's history in the sulphur industry goes back to the early 1950s. And while IPCO's entry into the drum granulation segment is relatively recent, its technological advancements have removed the barriers that have limited past drum granulation technologies, allowing the performance of IPCO drum granulators to surpass existing drum granulation processes.

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Alternative lean acid gas processing

New build economics for SRUs using Linde-Worley Parsons oxygen-enriched technology has been evaluated for applications in large gas plants processing lean acid gas containing benzene, toluene and xylenes (BTEX). Such plants typically require feed gas and/or combustion air preheating and the use of fuel gas co-firing and have not, historically, been considered for oxygen-enriched operation. **S. Pollitt** of WorleyParsons and **Dr M. Guzmann** of Linde Gas present the effects of oxygen enrichment in such cases and the resulting benefits in capital cost and operating cost reductions.

inde (also known as BOC) and WorleyParsons have worked together on the development and application of oxygen-enriched technology to SRUs for over 35 years. The basics of oxygen enrichment were explored using a custom-built pilot plant in the 1980s. The plant ran for three years and during that time work was done to fully characterise the operation of oxygen-enriched SRUs. The programme also resulted in the development of a burner for application in oxygen-enriched designs.

The results from the programme formed the basis of patents and licensed technology that has been jointly exploited by Linde and WorleyParsons in over 60 projects worldwide.

The advantages of SRU oxygen enrichment technology are well known to the industry:

- The replacement of air by oxygenenriched air or pure oxygen removes inert nitrogen from the system and allows more acid gas to be processed. At low and medium levels of enrichment this can result in a very low cost debottlenecking of the plant. As much as 50% increase in capacity can be achieved with little or no plant modification and up to 100% additional capacity can be achieved with plant modification, e.g. using Linde/WorleyParsons' SURE[™] double combustion technology.
- Operation of Claus units with enriched air or oxygen increases the tempera-

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ture in the Claus reaction furnace and ensures any contaminants such as aromatics and ammonia are completely destroyed. Such applications can reduce or eliminate the need for fuel gas co-firing in the furnace.

- The use of enriched air/oxygen expands the operating envelope of Claus units. The processing of lean acid gases can be achieved successfully without the need for additional heat sources or acid gas enrichment due to the achievement of stable flame temperature with lower H₂S concentrations
- The design of new-build Claus plants using oxygen will result in lower volume flows through the plant and reduced equipment sizes, resulting in reduced capital cost.

Historically the use of oxygen-enriched technology has been applied predominantly in the debottlenecking of existing SRUs where the economics of such projects is overwhelmingly positive. The maximum cost of such debottlenecking is typically 5-25% of the cost of building a new SRU train, depending on the enrichment technology selected.

It is for these reasons that oxygen enrichment has been used most often in the debottlenecking of existing refinery SRUs. Increases in capacity of over 30% are possible using "low level" enrichment (oxygen content up to 28% and higher depending on H_2S content in the feed gas) and increases in capacity of over 100% are achievable using pure oxygen.

New build SRUs using oxygen-enrichment are less prevalent and have tended to be built when a source of oxygen is readily available, such as the removal of sulphur from streams associated with gasification plants e.g. the Reliance petcoke gasification project which uses Linde/WorleyParsons SURE[™] technology.

The case study in this article addresses the new build economics for SRUs using oxygen enrichment in large gas plants processing lean acid gas, that is typical for gas processing plants.

Such designs have not been considered due to a number of perceived, but unquantified and thus often misjudged reasons:

- the (mis-)perceived cost of oxygen supply;
- the assumed unavailability of large oxygen volumes;
- the unknown simplicity and very high reliability of >99% oxygen supply even in remote locations at desulphurisation site with flexibility in energy supply;
- the (over-)estimated safety requirements associated with oxygen handling.

This article seeks to explore these perceived risks and present an economic and technical case for the consideration of oxygen enriched grassroots designs for large SRUs handling lean acid gas.

The effects on the plant design have been analysed and the life cycle costs

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compared with those of the conventional approach using air. The analysis focuses on a number of areas including: equipment size, oxygen supply, fuel gas consumption and power consumption.

Due to the smaller volumetric gas flow rate processed in oxygen-enriched plants SRU and tail gas treatment unit (TGTU) equipment will be smaller compared to conventional air design and in the case of multiple trains it can even reduce the number of trains required.

Supply of oxygen is an important part of the economic assessment. In this case study oxygen supply costs are considered as opex being supplied by an industrial gas company by building, investing and operating a dedicated, highly reliable air separation unit at customer site, thus not requiring capex.

When fuel gas co-firing is required in the reaction furnace for contaminant destruction, the use of oxygen enrichment reduces or eliminates co-firing requirements. There are also savings in fuel gas consumption in the thermal oxidiser downstream of the TGTU.

Power consumption is reduced due to the smaller equipment required for the same sulphur capacity. Savings are also seen due to the reduced air demand and size of the combustion air blower.

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Case study: rich gas vs lean gas application

The purpose of the study is to quantify the life cycle costs of oxygen-enriched SRUs compared with conventional Claus plants using air, for new SRUs at a scale commensurate with typical gas processing plants. The analysis also sought to identify the boundaries of any economic advantages of oxygen enrichment over traditional designs by considering a typical capacity for a gas processing plant and a range of H_2S concentrations.

The methodology adopted to achieve this was as follows:

- Identify the range of acid gas compositions that allows a broad and meaningful evaluation of the technologies.
- Develop process simulations, equipment sizing and utility consumptions for each case. Oxygen is accounted for in the analysis as a variable cost.
- The boundary for operation of the reaction furnace is set by the maximum permitted refractory temperature. In all cases considered this limit was not exceeded.
- Use the process simulations results to estimate capital, operating and maintenance costs to define a lifecycle cost comparison.

Cases evaluated

Different cases of oxygen concentration and H_2S concentrations were evaluated to ensure a complete range of feed conditions for the assessment.

Cases considered representative for the study were determined as follows:

- A sulphur plant capacity of 1,000 t/d was considered to be exemplary for the large gas plants single train design sizes.
- Acid gas feed concentration range was considered from 40 mol-% to 80 mol-% on a dry basis. The acid gas was simulated as saturated with water at 60°C and preheated to 240°C (HP steam preheater) before entering the reaction furnace in each case.
- BTEX was assumed to be present in all cases
- The minimum target temperature in the reaction furnace is taken as 1,100°C the temperature required to ensure complete destruction of BTEX.
- The 40% H₂S concentration case was studied but a viable solution purely based on oxygen enrichment was not possible. Even at 100% oxygen enrichment the target temperature of 1,100°C was not attainable. As such the 40% case would be limited to gas treatment only where no BTEX are present. Thus, this case has not been considered in this study.

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Table 1: Summary of KPIs

F		Flow rate from reaction furnace comparison		Air blowers power required comparisons (1)		Total fuel gas consumption comparisons (2)	
H ₂ S (vol-%)	Capacity (t/d)	Air only	Oxygen % of base	Air only	Oxygen % of base	Air only	Oxygen % of base
50	1,000	base	39.4	base	8.8	base	14.0
60	1,000	base	41.2	base	7.9	base	15.0
70	1,000	base	43.8	base	7.6	base	19.3
80	1,000	base	41.5	base	5.3	base	17.5

(1) Total combustion air required for reaction furnace. RGG and incinerator

(2) Fuel gas is total of RF, RGG and incinerator consumption, composition assumed to be: 94% methane, 2% ethane, 0.5% propane, 2.5% nitrogen, 1% CO2.

- The oxygen cases always consider 96% oxygen introduced in the reaction furnace. This limit is set by the reaction furnace refractory design temperature (1,760°C) and considers a safety margin.
- The oxygen is supplied at a concentration of 96% as this is assumed a cost effective level and minimises the oxygen supply cost. Oxygen is assumed to be supplied at 40°C and is not heated further before introduction into the furnace.
- Other impurities such as hydrocarbons or ammonia (normally only seen in refinery applications) in the acid gas feed were assumed not to be present in the feed gas.

Process description

Fig. 1 represents the process configurations for the cases with air only (shown in black) and with oxygen enrichment (modifications to air only configuration shown in red).

The inlet acid gas stream originates from an upstream gas treating regenerator and contains 40/50/60/70/80 vol-% H₂S (dry basis) with the balance being CO_2 . The feed gas is considered saturated with water at 60°C and contains only BTEX impurities.

The fuel gas composition assumed in the study is 95 vol-% methane, 2.5 vol-%, ethane and 2.5 vol-% nitrogen. Net heating value is assumed to be 47,000 kJ/kg.

Oxygen is provided at 40°C, 96 vol-% purity (balance nitrogen) at 0.8 barg, with no preheat. In all the oxygen cases no fuel gas co-firing is employed for safety reasons.

A comparison is made between a conventional design using air and an oxygen-enriched case where the maximum amount of oxygen is added while keeping within the temperature design limits of the

furnace refractory (in all cases considered here 96% oxygen enrichment is possible).

The configuration has two catalytic stages to recover approximately 94% of the sulphur in the feed gas, while the remainder is recovered in the tail gas unit to obtain a total sulphur recovery >99.9% in all the cases.

Fig. 1 highlights the places where oxygen allows savings in the plant: less air requires smaller air blowers, less power, less fuel gas and smaller equipment sizing overall for the plant.

To ensure consistency in the composition of tail gas recycled to the SRU the TGTU performance has been assumed to recover 99% of the tail gas $\rm H_2S$ (1% slipping) and 10% of the CO₂ (90% slipping) consistently with the behaviour of a highly selective H_2S amine based solvent that is particularly suited to large gas plants e.g. FLEXSORB®.

Simulation basis

Commercially available simulation software was used to model the various SRU cases. For the TGTU the simulation models the process stream up to the outlet of the direct contact condenser (DCC). The TGTU absorber/solvent regenerator has been consistently included in all cases and allows 99% H₂S and 10% CO₂ to be recovered in the recycled stream to the reaction furnace, while the remainder is fed to the off-gas incinerator.

Overall cost comparison

The process parameters that impact most significantly on the plant life cycle cost are:

• the processed gas volumetric flow rate, that affects the SRU-TGTU system equipment sizes (capex, opex) including off-sites, which are considered in the capex;

the power consumption (opex);

- the fuel gas consumption (opex);
- the oxygen consumption (opex). The • cost of oxygen provision was modelled as opex being supplied by industrial gas company.

Table 1 provides a summary of the key performance parameters between air only and pure oxygen options. Performance figures are indicated on a relative, rather than absolute basis, for the purpose of comparison, thus oxygen enrichment process parameters and utilities consumptions are expressed as percentages of air only case requirements.

Table 2 provides the cost comparison between the air cases and the oxygen enrichment cases: in all the cases oxygen reduces the overall volumetric gas flowrate to be treated in the SRU-TGTU system which translates in lower capital and operative costs for the equipment affected by the gas flow rate.

Performance figures are again indicated on a relative, rather than absolute basis, for the purpose of comparison, thus oxygen enrichment process parameters are expressed as percentages of air only case requirements.

Order of magnitude capital cost estimates were developed on a US Gulf Coast basis and include engineering (including technology license fees), equipment procurement and installation, catalyst and chemicals (including the SRU catalyst and TGTU solvent), plus indirect construction costs, spares and start-up services. In addition off-sites including the periphery of the SRU /TGTU are also considered in the capital costs. All estimates have been factored from a base case based on variation of key process flow rates.

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Table 2: Summary of cost comparison

		Capex		Opex		NPV of co	NPV of costs	
H ₂ S (vol-%)	Capacity (t/d)	Air only	Oxygen (% of base)	Air only	Oxygen (% of base)	Air only	Oxygen (% of base)	
50	1,000	base	57.2	base	74.3	base	63.7	
60	1,000	base	58.8	base	88.1	base	68.4	
70	1,000	base	60.9	base	103.4	base	72.9	
80	1,000	base	59.0	base	104.0	base	71.3	

In all cases the oxygen enrichment has

a beneficial impact on the capital cost as

it reduces the volumetric flow rate of gas

introduced in the SRU reaction furnace with

consequent size reduction of all the equip-

ment impacted by this flow. In general it

can be said that the lower the H₂S concen-

tration, the higher the beneficial impact of

the oxygen enrichment on the capital cost.

An exception can be seen in the 70% H_2S

concentration case. It shows an anomaly

as it has slightly lower capital cost bene-

fits than the other cases with a magnitude

of the deviation of < 2%. However, it can

be noted that this slight deviation can be

Fig. 3 shows the relative operative cost

comparison between air cases and the 96%

oxygen cases. As shown in the diagram the

operating costs are proportionally lower

when comparing leaner acid gas cases. For

the 80% H_2S and the 70% H_2S cases the

operating costs are slightly higher than the

ones from the air cases, as oxygen costs

are more evident through lower absolute

savings on other operating costs (i.e. fuel

attributed to simulation simplifications.

Operative cost

Operating and maintenance costs were determined based on the following general assumptions and operating cost basis:

acheran	
Discount rate:	10%
Project period:	20 years
Construction period:	3 years
Total SRU capacity:	1,000 t/d
Operating hours:	8,500 hours/year
Utilities value	
Power:	35.0 \$/MWh
Fuel gas:	10.35 \$/million kcal
	2.6 \$/million Btu
GOX:	0.07 \$/Nm ³
HP steam:	3.6 \$/t
Opex fixed	
0&M:	2.50 % of capex
Insurances:	0.30 % of capex
Overheads:	200.0 \$/t/d
Costs have been dis	scounted at a rate of
10% for each year of	an assumed 20 year
plant life, to determin	ne a Net Present Cost
for each case.	

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Fig. 2 represents the relative capital cost comparison between air cases (SRU-TGTU and off-sites) and the 96% oxygen cases (SRU-TGTU and off-sites)



ergies such as utilisation of the by-product nitrogen, that will reduce the oxygen cost and thus provide significant savings.

NPV of costs

Fig. 4 finally compares the actual net present cost (in million US\$) between the air cases and the 96% oxygen cases.

In all cases the net present cost of the oxygen enriched option is lower than the air only case. At lower H_2S concentrations the oxygen enrichment advantage is more pronounced than at higher concentrations due to the lower volumetric process gas flowrate which means smaller sized equipment and the lower operating costs as shown in the previous diagram. As already mentioned a small deviation can be seen at the 70% H_2S case based on slightly lower capital cost savings, which can be attributed to a simulation simplification.

In addition to the net present cost savings of the oxygen-enriched option through operating and capital cost savings, it can also be highlighted that the oxygen-enriched option shows less CO_2 emissions; the lower fuel gas consumption creates less CO_2 emissions with a magnitude of around 50,000 to 150,000 t/a, as shown in Table 3.

The lower limitation of the oxygen enrichment alternative is defined by the



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Fig. 2: Relative capex

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🔵 air case 🛑 oxygen case

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Table 3: CO₂ emission reduction (oxygen-enriched option)				
H ₂ S (vol-%)	Capacity (t/d)	CO_2 emission reduction (t/a)		
50	1,000	152,305		
60	1,000	96,563		
70	1,000	54,194		
80	1,000	48,219		

concentration of H_2S at which the use of pure oxygen is unable to achieve the required reaction furnace target temperature (in the study is fixed at 1,100°C as BTEX are assumed in the feed gas). However, this could be solved by introducing an acid gas enrichment unit (AGE) but has not been studied.

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The higher limitation for the use of oxygen enrichment technology is virtually the design temperature of the SRU reaction furnace refractory (1,760°C typically) but a safer and more realistic temperature limit is usually considered around 1,500°C. In all cases a single combustion reaction furnace is assumed, this leads to a maximum temperature of 1,594°C (80% H_2S in the feed gas and full oxygen enrichment). Technologies such as SURE[™] double combustion are available to safely handle these situations. In all cases studied, despite the additional cost of oxygen and the burner, the savings were significant.

Even at the high level of oxygen enrichment considered the furnace temperature limit is not exceeded in any case.

Fig. 5 represents the temperature in the reaction furnace for the 1,000 t/d case. As shown, the air case is kept in all cases above 1,100°C (to destroy BTEX contaminants) with the use of acid gas and air preheating as well as fuel gas co-firing (except for the 80% H_2S case that reaches 1,160°C even without the use of fuel gas). The 96% oxygen case shows instead a steady increase in the reaction furnace

Fig. 5: Reaction furnace temperature for 1,000 t/d case



temperature with the increasing H_2S feed gas concentration up to a maximum of 1,594°C for the 80% case.

Looking at H_2S concentration, net present cost savings are consistently high in all cases considered, due to the substantial operating cost savings (with exception for the 80% and 70% H_2S cases), capital cost savings associated with the equipment sizing and the relatively small impact of the cost of the oxygen.

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PUMPS

Vertical turbine pumps for sulphuric acid service

Pumping of sulphuric acid is a very demanding application, and the lifetime of the equipment is a challenge considering the severe corrosive conditions encountered in several parts of the process. Sulzer Pumps explains how the selection of the pump design is a fundamental step in obtaining the best operating performance of the equipment.

<caption>



luphuric acid (H_2SO_4) is one of the most important industrial chemicals as it is used in various process applications such as fertilizers, metals, pigments, explosives, and several others. The production of sulphuric acid is achieved through several steps. At first, liquid sulphur is burned in a furnace to produce sulphur dioxide (SO_2) . In this first phase, it is of primary importance to avoid humidity, which would lead to catastrophic corrosive conditions. The produced SO₂ gas is then processed by high-temperature catalytic oxidation to generate sulphur trioxide (SO_3) , which will be used in a contact process to produce sulphuric acid.

In a conventional contact phase process, depending on the installation, the concentration unit features absorption and drying towers, where an ascending stream of gas flows through a sprayed cloud of concentrated sulphuric acid. In the absorption tower, SO_3 reacts with H_2SO_4 and leads to a higher concentration of sulphuric acid, which is then diluted by the addition of water. This process requires continuous pumping of sulphuric acid from a tank to the tower. The principle of the drying tower is similar, but it uses ambient air instead of SO_3 gas. It is an important operation since sulphuric acid droplets capture the humidity of air so that the dried air is then suitable to be injected into the sulphur burning furnace.

In most configurations, vertical pumps are installed at the top of a tank, ensuring maximum safety and reliability. This layout does not require the connection of the pump to the tank under the liquid level, and any leakage that may occur will be contained. Additionally, it ensures safer on-site installation and maintenance because those operations are performed above the tank.

The typical operating parameters for circulation pumps used in the above application are:

- Liquid: H_2SO_4
- Concentration: 92-99.9%
- Temperature: 60-120°C (158-248°F)
- Flow: up to $1,800 \text{ m}^3/\text{h} (7,800 \text{ g/m})$
- Head: 15-30 m (49-99 ft)

More stringent conditions could be managed with an alternative process design. Due to exothermic reactions, the global heat generated in the process is important. The most common plant design uses this energy to generate low-temperature steam, which is then transformed into electricity. Process licensors have developed heat

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recovery systems allowing the production of higher-temperature steam, which is more efficient in electricity production. This is achieved by using a similar tower working at a much higher temperature than in the conventional process.

The typical operating parameters for heat recovery circulation pumps used in the above application are:

• Liquid: H_2SO_4

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- Concentration: 99.9%
- Temperature: 200-250°C (392-482°F)

The challenge

Sulphuric acid is highly corrosive, and a variation of concentration or temperature could increase corrosion drastically, resulting in considerable damage to the plant equipment. The challenge for operators is to keep those parameters under control, and this is more difficult to achieve during transient phases (i.e. starting, shut-off, surge, etc.) than other phases. For that reason, equipment lifetime and reliability are some of the most important factors to limit the number of those difficult phases and related risks.

Considering pump operation, the two main concerns would be material corrosion and mechanical issues. Mechanical issues (i.e. bearing lifetime, wear part consumption) are the same for corrosive applications and non-corrosive applications, while both are not totally independent since corrosion could affect the geometry of parts and have subsequent effects increasing mechanical issues. In the worst conditions, it could turn into an exponential phenomenon with catastrophic damage to the pump. The challenge for pump manufacturers is to select a design that shows the best performance in those conditions.

Design assessment

In the present case, the two main primary characteristics to define the pump design are the material of construction and the pump type itself. The material selection requires good metallurgical and process knowledge since the corrosiveness of sulphuric acid varies greatly depending on the concentration and temperature. High-concentration 99% sulphuric acid at a medium temperature of 70°C may be pumped with a pump made of acid-proof cast iron materials, while reducing to a lower concentration below 94% may require a higher-grade alloy.

Material performance may be assessed with corrosion rates available in literature.



On the other hand, it could be difficult to find similar operating conditions that correspond to the actual plant operating conditions. Consequently, material selection is achieved through a complex process based on academic research, laboratory testing, and most importantly considering the field experience of users and the manufacturer. Meanwhile, most data or field tests are available for static conditions only. Knowing that flow velocity has an impact on erosion, the corrosion rate of the material eventually varies. In some cases, a material with the lowest corrosion rate in static conditions has shown a higher corrosion rate in dynamic conditions.

Considering the observation above, the challenge is then to select the vertical pump design that gives the best intrinsic performance independently of material selection. The two main designs that are available for vertical pumping out of a tank are verticallymounted end suction pump (Fig. 1) or vertical turbine pump (Fig. 2). The first one is the most common for these applications to date. It features a volute case installed on a suspension column with a separate discharge pipe rising up to a baseplate. The volute case geometry generates radial thrust on the pump line shaft. The thrust leads to deflection and vibrations and causes wear of the pump bush bearing and the roller bearings. As previously mentioned, the highly corrosive conditions, where the clearance increases over time, further deteriorates the mechanical condition of the pump. This effect being exponential, the equipment lifetime is consequently rapidly decreased.

In a vertical turbine pump, the medium is pumped directly through the impeller from the suction to the column and discharge head. The symmetric diffuser case (Fig. 3) of the pump distributes the thrust equally. Detrimental thrust on the line shaft does not occur and as a result, the level of



vibrations and the shaft deflection can be kept at a minimum. This advantage does not only apply to the best efficiency point of the pump, but to the entire flow range.

A radial thrust comparison (Fig. 4) clearly shows the radial thrust variation depending on the case design. A single volute shows the worst impact. In all cases, a vertical turbine pump design keeps radial thrust at the lowest value, further preventing mechanical damage to the pump. In the presence of a high temperature, the vertical turbine pump design with only the discharge column filled with liquid does not present a risk of stress due to the differential thermal expansion of an asymmetric construction with the suspension column partially filled with liquid.

Another aspect to be considered is the localised corrosion observed at the acid/ gas interface; the result of localised liquid concentration and temperature. Devastating consequences could arise due to that phenomenon. Good plant maintenance practice requires metal thickness measurements to monitor the condition of the parts and ensure their replacement in due time if necessary. A vertically-mounted pump features four interfaces (i.e. two on the support column, one on the shaft and one on the discharge pipe), while a vertical turbine pump only has one interface on the discharge column. This limits corrosion, making monitoring easier and ultimately reducing the risk of catastrophic failure significantly.

The compact design of a vertical turbine pump gives additional benefits for installation and maintenance. Discharge through a column and sump head requires a footprint dimension which is less than half of that needed by a volute case design. A side discharge on the sump head can be flanged directly to a downstream discharge pipe. Additionally, pump assembly is much

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easier with only one column assembly. As a result of this assessment, the VAS pump type (Fig. 5) has been designed combining all of the advantages of the vertical turbine pump with adequate material selection and technical adjustments adapted to the specific requirements of the process. It benefits from all of the experience accumulated while developing and manufacturing heavy duty equipment for demanding applications.

The current range performance covers flow rates up to 4,000 m³/h, and higher flow rates could be considered based on extended standard vertical turbine pump flow range (Fig. 6). The standard construction is suitable for absorption tower and drying tower circulation, and operation in a heat recovery system requires specific features to ensure safe operation at higher temperatures.

Experience

The original VAS design was made back in the 1970's by Ensival, a Belgian company that subsequently merged into Ensival Moret



and was eventually acquired by Sulzer in 2017. The company was working actively to develop and manufacture a pump for severe industrial applications. Capitalising on the company's huge know-how and field experience of highly corrosive applications, combined with local engineering collaboration, the pump type has been operating since that period with high user satisfaction. Equipment has been supplied for numerous projects worldwide either as original supplies for new plants or as a replacement of other manufacturers' equipment, with a high level of user satisfaction. Depending on the operating conditions of the plant, the overall lifetime of the pumps has been four to eight years with limited spare parts consumption. The company is continuously supporting users to improve their existing equipment or to allow process developments as illustrated by recent projects.

Case study 1

A leading global fertilizer producer was facing difficulties in optimising the operation in one of its sulphuric acid plants because of limited pumping equipment lifetime and increased process downtime. The equipment involved was a circulation pump in a heat recovery system. The lifetime of the pump originally supplied with the system was less than one year and consequently far out of plant standards. Being the sole installed running pump, troubleshooting of the pump required system shut-down.

As a satisfied user of VAS pumps for decades, the plant team contacted our technical department to discuss the problem faced with a competitor's equipment. Sharing our expertise in pump design and materials for sulphuric acid pumping, we have helped the customer increase the equipment lifetime slightly. Meanwhile, problems due to radial thrust, which were clearly visible on damaged parts, were intrinsic to the volute case design. The existing pump was eventually replaced by our vertical turbine pump design, which has now demonstrated more than a double lifetime compared to the previous equipment at the customer's site. The pump is still running to date, so the customer is now planning to extend the operating period between system shut-downs.

Case study 2

A current development trend for designing sulphuric acid plants require an increase in the pump flow. The largest existing plant currently in operation features a continuous circulation of two pumps with a typical flow between 1,200 m³/h and 1,800 m³/h. An engineering company identified the benefits of operating a single pump designed for the total capacity (i.e. tank lay-out, single downstream line, etc.). The initial requirement considered a single pump in operation with a flow rate of 2,600 m³/h.

To cope with this significant performance increase, the equipment size should be much larger, amplifying the disadvantage of a vertically-mounted end suction pump. Due to the asymmetric volute case design, the radial thrust value and thermal stress would have more than a significant impact, drastically limiting the equipment lifetime. On the other hand, a vertical turbine pump does not present those technical issues, whichever the equipment size. Considering those benefits, the project has proceeded with a single pump for a sulphuric acid flow of 2,800 m³/h.

Additionally, a compact design avoids the oversizing of the equipment and allows easy maintenance and installation as demonstrated on the test bench during the manufacturing process.

Conclusions

Sulzer's VAS vertical turbine pump type has been specifically designed for sulphuric acid pumping, and has helped users improve operation performance considerably even in the most stringent conditions. The pump design extends the lifetime of the equipment and allows the customer to increase the mean time between maintenance significantly. The compact design of the vertical turbine pump ensures easy installation and maintenance-friendly operation, while the symmetric diffuser case design allows the development of bigger pumps for higher sulphuric acid circulation flows than with existing equipment, opening new paths for process development.

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